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NOTES ON QUALITATIVE ANALYSIS

CONCISE AND EXPLANATORY.

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NOTES ON

QUALITATIVE ANALYSIS

CONCISE AND EXPLANATORY

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PREFACE TO THE THIRD EDITION.

In revising the present edition, a few minor corrections and alterations have been made in matters of detail, and some additional foot-notes added. Beyond these I have not considered it necessary or advisable to make any material change in the scope or arrangement of the book.

H. J. H. F.

January, 1888.

PREFACE TO THE FIRST EDITION.

The principal object of the present work, while setting forth the usual methods of Qualitative Analysis in their most concise form, is to direct, and if possible to enforce, attention to the rationale of each operation performed, and the nature of each reaction which takes place.

It is hoped that the system adopted will tend to some extent to check the prevailing tendency, common even among more advanced students, to regard Chemical Analysis as a mere routine of mechanical operations, and entirely to overlook its scientific aspect.

It has been my endeavour in drawing up the Analytical Tables, with their Explanations, to render each one comprehensive, avoiding as far as possible the necessity of reference to remote sections or pages.

The scheme of analysis recommended is in substance the same as that given in the standard text-books, but considerable changes have been introduced in many important matters of detail, and in arrangement, such as experience has shewn to be advantageous.

The reactions of some of the more important Organic Substances have been stated in a short form, to meet the requirements of commencing Medical Students.

I am greatly indebted to my friend W. J. SELL, M.A., for his kindness in revising the proof-sheets, and in making some useful suggestions.

H. J. H. F.

April, 1883.

PREFACE TO THE SECOND EDITION.

BUT few alterations or additions have been made in the present edition of these Notes. They have been exhaustively tested in the hands of many hundreds of students of various classes, and have been found satisfactorily to fulfil the objects with which they were written. It has been suggested that the detection and reactions of the "rare" elements should be included; but such would be quite without the scope or intention of the work. It appears to me that in the so-called "common" elements we have ample material—if not too much—wherewith to set forth the principles of Qualitative Analysis on a scientific basis.

Much has been written and said of late calling in question the utility of Qualitative Analysis as a method of scientific training. If the object be solely the identification of unknown substances, and the directions for so doing consist merely of a sequence of bare recipes—like those in a cookery book—it is probable that it has but little value in this respect. But if, while keeping the analytical object ostensibly in view, the processes be employed as means of bringing the student face to face with, and fixing his attention on, various important types and characters of chemical change, Qualitative Analysis becomes a most useful aid in the scientific teaching of Chemistry. The operations are, for the most part, easily performed and successful even in the hands of a beginner. It is of course admitted that these desirable objects may be more directly attained by a series of specially devised experiments, but such are, as a rule, without the reach of the average teacher and student.

January, 1886.

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PRINCIPAL REACTIONS OF THE MORE COMMON METALS.

SILVER.

Solutions of silver salts (*e.g.* $AgNO_3$) give with

Hydrochloric acid, white ppt. of silver chloride soluble in NH_3, insoluble in HNO_3,

$$AgNO_3 + HCl = AgCl + HNO_3.$$

This precipitate is turned violet on exposure to sunlight.

Potassium chromate, crimson ppt. of silver chromate, soluble in HNO_3,

$$2AgNO_3 + K_2CrO_4 = Ag_2CrO_4 + 2KNO_3.$$

Caustic potash, brown ppt. of silver oxide Ag_2O, soluble in NH_3 and in acids.

Potassium iodide, yellow ppt. of silver iodide AgI, insoluble in NH_3 and in dil. HNO_3.

Potassium Cyanide, white ppt. of silver cyanide AgCy, soluble in NH_3 and in excess of KCy, insoluble in dil. HNO_3.

Sodium phosphate, yellow ppt. of silver phosphate Ag_3PO_4, soluble in NH_3 and in acids.

Hydrosulphuric acid, black ppt. of silver sulphide Ag_2S, soluble in hot HNO_3

Heated on charcoal, before the blow-pipe, with Na_2CO_3, solid silver compounds give brilliant white metallic mass or globules.

LEAD.

Solutions of lead salts, *e.g.* $Pb(NO_3)_2$, give with

Hydrochloric acid, white ppt. of lead chloride, soluble in boiling water, insoluble in ammonia,

$$Pb(NO_3)_2 + 2HCl = PbCl_2 + 2HNO_3.$$

Hydrosulphuric acid, black ppt. of lead sulphide*,

$$Pb(NO_3)_2 + H_2S = PbS + 2HNO_3.$$

This ppt. dissolves in hot *dilute* HNO_3, forming lead nitrate. *Strong* nitric acid converts it chiefly into white insoluble lead sulphate.

Dilute sulphuric acid, white ppt. of lead sulphate, soluble in strong solution of ammonium acetate,

$$Pb(NO_3)_2 + H_2SO_4 = PbSO_4 + 2HNO_3.$$

Potassium chromate, yellow ppt. of lead chromate, insoluble in acetic acid, soluble in KHO,

$$Pb(NO_3)_2 + K_2CrO_4 = PbCrO_4 + 2KNO_3.$$

Potassium iodide, yellow ppt. of lead iodide PbI_2.
Caustic potash, white ppt. of hydrated lead oxide $2PbO . H_2O$, soluble in excess.
Ammonia, white ppt. of basic salt, insoluble in excess.

Solid lead compounds heated on charcoal before the blow-pipe give yellow or orange incrustation, and, in the reducing flame, with Na_2CO_3, metallic beads which are malleable.

---

* If much HCl is present the ppt. is often dark red and consists of $PbS . PbCl_2$.

MERCURY.

General Reactions.

Solutions of mercury compounds heated with metallic copper and dilute hydrochloric acid (free nitric acid must be absent) give a bright deposit of metallic mercury on the copper. On heating the two metals in a dry narrow test-tube, the mercury sublimes, forming a grey ring which may be united into globules by rubbing with a glass rod.

Solid mercury compounds heated on charcoal volatilize entirely. Heated in a bulb-tube with dry Na_2CO_3 a ring of metallic mercury is obtained.

Mercurous Salts.

Solutions of mercurous salts, *e.g.* $Hg'_2(NO_3)_2$, give with

Hydrochloric acid, white ppt. of mercurous chloride,

$$Hg_2(NO_3)_2 + 2HCl = Hg_2Cl_2 + 2HNO_3.$$

7

This ppt. is blackened by ammonia, owing to the formation of dimercurous-ammonium chloride—

$$Hg_2Cl_2 + 2NH_3 = (NHg'_2H_2)Cl \overset{+Hg}{+} NH_4Cl.$$

Caustic potash, black ppt. of mercurous oxide, insoluble in NH_3,

$$Hg_2(NO_3)_2 + 2KHO = Hg_2O + 2KNO_3 + H_2O.$$

Ammonia, black ppt. of a basic mercurous salt.

Potassium iodide, greenish-yellow ppt. of mercurous iodide Hg_2I_2.

Hydrosulphuric acid, black ppt. of mercurous sulphide Hg_2S (perhaps a mixture of $HgS + Hg$).

Stannous chloride, ferrous sulphate, or *sulphurous acid*, grey ppt. of metallic mercury.

Potassium chromate, orange ppt. of basic mercurous chromate $Hg_2CrO_4 . Hg_2O$.

Mercuric Salts.

Solutions of mercuric salts (*e.g.* $Hg''Cl_2$) give with

Hydrosulphuric acid, black ppt. of mercuric sulphide,

$$HgCl_2 + H_2S = HgS + 2HCl.$$

This ppt. is insoluble in ammonium sulphide and in dilute HNO_3. Strong HNO_3 converts it into a white mixture of $2HgS$ and $Hg(NO_3)_2$.

Caustic potash, yellow ppt. of mercuric oxide,

$$HgCl_2 + 2KHO = HgO + 2KCl + H_2O.$$

Potassium iodide, red ppt. of mercuric iodide HgI_2, soluble in excess, forming the double iodide ($HgI_2 . 2KI$).

Ammonia, white ppt. of mercuric-ammonium chloride ($NHg''H_2)Cl$.

Stannous chloride, first a white ppt. of mercurous chloride, which is afterwards further reduced to metallic mercury, if sufficient $SnCl_2$ is present.

COPPER.

Cupric Salts (usually green or blue).

Solutions of cupric salts (*e.g.* $CuSO_4$) give with

Hydrosulphuric acid, black ppt. of cupric sulphide,

$$CuSO_4 + H_2S = CuS + H_2SO_4.$$

This ppt. is nearly insoluble in ammonium sulphide. Soluble in hot dilute HNO_3, forming cupric nitrate $Cu(NO_3)_2$. Insoluble in dilute H_2SO_4. [The precipitation by H_2S is prevented by the presence of potassium cyanide owing to the formation of a double cyanide $Cu_2Cy_2 . 2KCy$.]

Potassium ferrocyanide, chocolate ppt. of cupric ferrocyanide,

$$2CuSO_4 + K_4(FeCy_6) = Cu_2(FeCy_6) + 2K_2SO_4.$$

Ammonia, pale greenish-blue ppt. of a basic cupric sulphate (such as $CuSO_4 . 2Cu(OH)_2$), readily soluble in excess of NH_3, forming a dark blue solution. This solution contains a compound having the composition $(CuSO_4 + 4NH_3 + H_2O)$ or $\{(N_2H_6Cu)O + (NH_4)_2SO_4\}$.

Caustic potash, pale blue ppt. of cupric hydroxide $Cu(OH)_2$, which turns black on boiling, CuO being formed.

Potassium iodide, dirty white ppt. of cupr*ous* iodide Cu_2I_2, free iodine being liberated.

Potassium cyanide, brownish-yellow or olive-green ppt. of cupric cyanide $Cu''Cy_2$, which rapidly loses cyanogen, becoming $(2Cu''Cy_2 . Cu'_2Cy_2)$ or Cu'_2Cy_2.

These cyanides dissolve in excess of KCy, forming double cyanides $CuCy_2 . 2KCy$ and $Cu_2Cy_2 . 2KCy$ respectively.

Many *reducing agents* such as glucose, aldehyde, &c. in presence of caustic potash, precipitate red cuprous oxide Cu_2O, on heating.

Cuprous Salts (usually white, and insoluble in water).

Solutions of cuprous salts (*e.g.* Cu_2Cl_2 dissolved in HCl) give with

Caustic potash, yellow ppt. of cuprous hydroxide $Cu_2(OH)_2$.

Potassium iodide, white ppt. of cuprous iodide *without* separation of iodine,

$$Cu_2Cl_2 + 2KI = Cu_2I_2 + 2KCl$$

Water gives a white ppt. of Cu_2Cl_2, this being soluble only in *strong* HCl.

Solid copper compounds heated with Na_2CO_3 on charcoal before the blow-pipe give red globules or spangles of metallic copper.

Borax bead, in oxidizing flame, green when hot, blue when cold.

CADMIUM.

Solutions of cadmium salts (*e.g.* $CdCl_2$) give with

Hydrosulphuric acid, bright yellow ppt. of cadmium sulphide,

$$CdCl_2 + H_2S = CdS + 2HCl.$$

This ppt. is insoluble in ammonium sulphide and in potassium cyanide. Soluble in hot dilute HNO_3 forming cadmium nitrate $Cd(NO_3)_2$. Soluble in dilute H_2SO_4.

Caustic potash, white ppt. of cadmium hydroxide, insoluble in excess,

$$CdCl_2 + 2KOH = Cd(OH)_2 + 2KCl.$$

Potassium cyanide, white ppt. of cadmium cyanide,

$$CdCl_2 + 2KCy = CdCy_2 + 2KCl.$$

This ppt. dissolves in excess forming $CdCy_2 . 2KCy$ from which solution H_2S precipitates cadmium sulphide.

Ammonia gives same ppt. as caustic potash (from a sufficiently strong solution) easily soluble in excess.

Solid cadmium compounds heated on charcoal in the reducing flame with Na_2CO_3 give a brown incrustation.

BISMUTH.

Solutions of bismuth salts (*e.g.* $Bi(NO_3)_3$), give with

Hydrosulphuric acid, blackish-brown ppt. of bismuth sulphide,

$$2Bi(NO_3)_3 + 3H_2S = Bi_2S_3 + 6HNO_3.$$

This ppt. is insoluble in ammonium sulphide. Soluble in hot dilute HNO_3, forming bismuth nitrate $Bi(NO_3)_3$.

Ammonia, white ppt. of bismuth hydroxide,

$$Bi(NO_3)_3 + 3NH_3 + 3H_2O = Bi(OH)_3 + 3NH_4NO_3.$$

This ppt. dissolves in HCl, forming bismuth chloride $BiCl_3$. Much water added to the solution precipitates white bismuth oxychloride $Bi_2O_3 . BiCl_3$,

$$3BiCl_3 + 3H_2O = Bi_2O_3 . BiCl_3 + 6HCl.$$

Bismuth oxychloride dissolves in hydrochloric but not in tartaric acid.

Stannous chloride + caustic potash, black ppt. of bismuth dioxide Bi_2O_2.

Potassium chromate, yellow ppt. of basic bismuth chromate $3Bi_2O_3 . 2CrO_3$, insoluble in KHO, soluble in dilute HNO_3.

Potassium iodide, brown ppt. of bismuth iodide BiI_3, soluble in excess.

Caustic potash, white ppt. of bismuth hydroxide $Bi(OH)_3$.

ARSENIC.

General Reactions.

Compounds of arsenic in solution acidified with HCl (free HNO_3 should be absent) and introduced into a vessel in which hydrogen is being generated (*e.g.* by the action of Zn + dil. H_2SO_4), give off arseniuretted hydrogen AsH_3, which burns with a bluish-white flame. If a piece of cold porcelain be held in the flame, a black stain of metallic arsenic is deposited on it. This stain dissolves at once in *sodium hypochlorite*. [Marsh's test.]

[AsH_3 is also slowly evolved if Zn and KOH (strong sol.) are used instead of Zn and H_2SO_4.]

If arseniuretted hydrogen be passed into a solution of silver nitrate, it is decomposed as follows:

$$AsH_3 + 6AgNO_3 + 3H_2O = \underset{\text{black ppt.}}{6Ag} + H_3AsO_3 + 6HNO_3.$$

On carefully neutralizing the clear liquid with ammonia (and adding a drop more $AgNO_3$, if necessary), a yellow ppt. of silver arsenite is obtained (see below).

Solid arsenic compounds heated on charcoal with Na_2CO_3 (or KCy) in the reducing flame give a characteristic (poisonous) odour of garlic.

Heated in a bulb-tube with Na_2CO_3 and charcoal, a black ring of metallic arsenic is obtained. This ring when heated in a dry test-tube gives a crystalline sublimate of arsenic trioxide As_2O_3.

Arsenious Compounds.

Solutions of arsenious compounds (*e.g.* K_3AsO_3) give with

Hydrosulphuric acid in presence of dilute HCl, yellow ppt. of arsenious sulphide,

$$2K_3AsO_3 + 6HCl + 3H_2S = As_2S_3 + 6KCl + 6H_2O.$$

This ppt. dissolves in *ammonium sulphide*, forming an ammonium sulpharsenite, *e.g.* $(NH_4)_3AsS_3$ (or $3(NH_4)_2S \cdot As_2S_3$); and in *ammonium carbonate* giving a mixture of arsenite and sulpharsenite. From either of these solutions dilute acids reprecipitate As_2S_3, thus:

$$2\underbrace{(NH_4)_3AsS_3}_{\text{sulpharsenite}} + 6HCl = As_2S_3 + 6NH_4Cl + 3H_2S$$

and

$$\underbrace{(NH_4)AsO_3}_{\text{arsenite}} + 3\underbrace{(NH_4)AsS_3}_{\text{sulpharsenite}} + 4HCl = 2As_2S_3 + 4NH_4Cl + 2H_2O.$$

Silver nitrate, yellow ppt. of silver arsenite, soluble in ammonia and in acids,

$$K_3AsO_3 + 3AgNO_3 = Ag_3AsO_3 + 3KNO_3.$$

Copper sulphate, yellowish-green ppt. of cupric hydrogen arsenite $CuHAsO_3$, which, when heated with caustic potash, gives red cuprous oxide Cu_2O.

Magnesium sulphate, in presence of NH_4Cl and NH_3, no ppt.

Metallic copper, in presence of dilute HCl, steel-grey deposit of copper arsenide, which, when heated in an open tube, gives crystalline sublimate of As_2O_3.

Oxidizing agents, such as nitric acid, sodium hypochlorite, &c., convert arsen*ious* compounds into the arsen*ic* form.

Arsenic Compounds.

Solutions of arsenic compounds (*e.g.* K_3AsO_4) give with

Hydrosulphuric acid, in presence of dilute HCl, on heating, at first, white ppt. of sulphur,

$$K_3AsO_4 + 3HCl + H_2S = H_3AsO_3 + 3KCl + H_2O + S,$$

and, after some time, yellow ppt. of As_2S_3,

$$2H_3AsO_3 + 3H_2S = As_2S_3 + 6H_2O.$$

[If first boiled with SO_2, As_2S_3 is at once precipitated (see below).]

Silver nitrate, brick-red ppt. of silver arsenate,

$$K_3AsO_4 + 3AgNO_3 = Ag_3AsO_4 + 3KNO_3,$$

soluble in ammonia and in acids.

Copper sulphate, greenish-blue ppt. of cupric hydrogen arsenate $CuHAsO_4$ (not reduced on heating with KHO).

Magnesium sulphate + ammonia, in presence of NH_4Cl, white crystalline ppt. of magnesium ammonium arsenate $Mg(NH_4)AsO_4$.

Ammonium molybdate, in presence of HNO_3, yellow ppt. on heating.

Ferric chloride, yellowish-white ppt. of ferric arsenate $FeAsO_4$.

Calcium chloride, white ppt. of calcium arsenate $Ca_3(AsO_4)_2$.

Many *reducing agents*, such as SO_2, convert arsen*ic* compounds into the arsen*ious* form

$$As_2O_5 + 2SO_2 + 2H_2O = As_2O_3 + 2H_2SO_4.$$

ANTIMONY.

General Reactions.

Compounds of antimony in solution acidulated with HCl, and poured on to a piece of *zinc* in contact with *platinum* foil, give a black stain of metallic antimony *on the platinum*.

In *Marsh's apparatus* they behave similarly to compounds of arsenic, but the black stain produced does not dissolve in sodium hypochlorite, [nor is SbH_3 evolved if Zn and KOH be employed].

Antimoniuretted hydrogen SbH_3 passed into a solution of *silver nitrate* is decomposed as follows:

$$SbH_3 + 3AgNO_3 = \underbrace{SbAg_3}_{\text{black ppt.}} + 3HNO_3.$$

The Sb may be dissolved out of this ppt. with tartaric acid, and confirmed by $HCl + H_2S$, as below.

Solid antimony compounds heated on charcoal before the blow-pipe with Na_2CO_3, in the reducing flame, give brittle grey metallic globules, and a white incrustation of Sb_2O_3.

Antimonious Compounds.

Solutions of antimonious compounds (*e.g.* $SbCl_3$) give with

Hydrosulphuric acid (in presence of dilute HCl), orange ppt. of antimonious sulphide,

$$2SbCl_3 + 3H_2S = Sb_2S_3 + 6HCl.$$

This ppt. dissolves in *ammonium sulphide*, forming an ammonium sulph-antimonite, *e.g.* $(NH_4)_3SbS_3$, from which it is reprecipitated by dilute acids; and in strong boiling HCl, forming antimonious chloride $SbCl_3$. It is almost insoluble in ammonium carbonate.

Silver nitrate, in presence of caustic potash, black ppt. of argentous oxide (?) which is *insoluble in ammonia*,

$$SbCl_3 + 4AgNO_3 + 8KHO = Ag_4O\ (?) + KSbO_2 + 4KNO_3 + 3KCl + 4H_2O.$$

Water gives white ppt. of antimonious oxychloride $Sb_2O_3 . SbCl_3$, soluble in *tartaric acid* and in HCl. HCl gives a similar ppt. from many antimonious compounds (*e.g.* tartar emetic) readily soluble in excess.

Caustic potash, white ppt. of antimonious oxide Sb_2O_3, soluble in excess, forming potassium antimonite $K_2O . Sb_2O_3$.

Ammonia gives same ppt. as KHO, nearly insoluble in excess.

Potassium iodide + HCl gives no iodine on heating.

Antimonic Compounds.

Solutions of antimonic compounds (*e.g.* $KSbO_3 = K_2O . Sb_2O_5$) give with

Hydrosulphuric acid, in presence of dilute HCl, orange ppt. consisting chiefly of antimonic sulphide,

$$K_2O . Sb_2O_5 + 2HCl + 5H_2S = Sb_2S_5 + 2KCl + 6H_2O.$$

This ppt. dissolves in *ammonium sulphide*, forming ammonium sulph-antimoniate $(NH_4)_3SbS_4$, from which it is reprecipitated by dilute acids; and in strong boiling HCl, forming antimoni*ous* chloride and free sulphur,

$$Sb_2S_5 + 6HCl = 2SbCl_3 + 3H_2S + S_2.$$

Heated with *potassium iodide* and *hydrochloric acid*, iodine is set free:

$$KSbO_3 + 6HCl + 2KI = SbCl_3 + 3KCl + 3H_2O + I_2.$$

The precipitate produced by *silver nitrate* and *caustic potash* is *soluble* in ammonia.

These last two reactions distinguish antimon*ic* from antimoni*ous* salts.

TIN.

General Reactions.

If a small piece of zinc be added to a solution containing tin acidulated with dilute HCl, a grey deposit of metallic tin is obtained on the zinc. On dissolving this deposit (with the zinc, if necessary) in strong hot HCl, stann*ous* chloride is obtained, which may be confirmed, as below, with mercuric chloride.

Solid tin compounds heated on charcoal with Na_2CO_3 and KCy in the reducing flame give malleable beads of metallic tin. Oxides of tin moistened with cobalt nitrate and heated in the oxidizing flame give a bluish-green mass.

Stannous Salts.

Solutions of stannous salts (*e.g.* $SnCl_2$) give with

Hydrosulphuric acid, in presence of dilute HCl, dark brown ppt. of stannous sulphide,

$$SnCl_2 + H_2S = SnS + 2HCl.$$

This ppt. dissolves in *yellow* ammonium sulphide[*], forming ammonium sulphostann*ate* $(NH_4)_2SnS_3 [= (NH_4)_2S . SnS_2]$ from which dilute HCl precipitates dirty yellow stann*ic* sulphide SnS_2. Stannous sulphide is soluble in strong boiling HCl, forming stannous chloride. Insoluble in *ammonia*.

[*] The yellow "ammonium sulphide" used in laboratories contains an excess of sulphur, being a mixture of polysulphides of ammonium, ammonium thiosulphate, &c.

Stannous Salts (*continued*).

Mercuric chloride, white ppt. of mercurous chloride, turning to grey metallic mercury if sufficient stannous salt is present, especially on heating,

$$SnCl_2 + 2HgCl_2 = Hg_2Cl_2 + SnCl_4$$
$$SnCl_2 + Hg_2Cl_2 = 2Hg + SnCl_4.$$

Auric chloride, purple ppt. of aurous stannous stannate (purple of Cassius) $Au'_2Sn''(SnO_3)_3$ (or perhaps $Au_2 + 3SnO_2$).

Caustic potash, white ppt. of hydrated stannous oxide $SnO . 2H_2O$, soluble in excess, forming potassium stannite $K_2SnO_2 (= K_2O . SnO)$.

Ammonia, same ppt. as KHO, insoluble in excess.

Stannous salts act as reducing agents, becoming converted into the stannic form; thus *potassium chromate* or *permanganate* (in acidulated solution) are at once reduced, giving a green chromium salt, and a colourless manganous salt respectively. Silver nitrate gives a black ppt. of metallic silver. Ferric and cupric salts are reduced to the ferrous and cuprous state.

Stannic Salts.

Solutions of stannic salts (*e.g.* $SnCl_4$) give with

Hydrosulphuric acid, in presence of dilute HCl, dirty yellow ppt. of stannic sulphide,

$$SnCl_4 + 2H_2S = SnS_2 + 4HCl.$$

This ppt. dissolves in *ammonium sulphide*, forming ammonium sulphostannate $(NH_4)_2SnS_3$, from which HCl reprecipitates SnS_2. Soluble in strong boiling HCl, forming stannic chloride. Slightly soluble in *ammonium carbonate*. Soluble in *ammonia*.

Mercuric chloride, no ppt. (Distinction from stann*ous* salts.)

Many *neutral salts* (*e.g.* Na_2SO_4 or KNO_3) give, in dilute solutions, on heating, white ppt. of metastannic acid $H_2Sn_5O_{11} . 4H_2O (= 5SnO_2 . 5H_2O)$.

Caustic potash, white ppt. of hydrated stannic oxide $SnO_2 . H_2O$, soluble in excess, forming potassium stannate $K_2SnO_3 (= K_2O . SnO_2)$.

Ammonia, a similar ppt. incompletely soluble in large excess.

Stannic salts may be reduced to the stannous form by *metallic copper* or *tin*, or by reducing to metal by *zinc* and dissolving in strong HCl.

IRON.

Ferrous Salts.

Solutions of ferrous salts (*e.g.* $FeSO_4$) give with

Ammonia, dirty green ppt. (white, if pure) of ferrous hydroxide,

$$FeSO_4 + 2NH_3 + 2H_2O = Fe(OH)_2 + (NH_4)_2SO_4.$$

This precipitation is partly prevented by NH_4Cl. The ppt. rapidly absorbs oxygen from the air, becoming reddish-brown hydrated Fe_2O_3.

Potassium ferricyanide, dark blue ppt. of ferrous ferricyanide (?) "Turnbull's blue,"

$$3Fe''SO_4 + K_6(Fe_2Cy_{12})^{vi} = Fe''_3(Fe'''_2Cy_{12})^{vi} + 3K_2SO_4 *.$$

Potassium ferrocyanide, pale blue ppt. of ferrous potassium ferrocyanide (white, if quite pure),

$$FeSO_4 + K_4(FeCy_6) = FeK_2(FeCy_6) + K_2SO_4.$$

Nitric acid, *potassium permanganate*, *potassium bichromate*, and other oxidising agents, convert ferr*ous* into ferric compounds,

$$6FeSO_4 + 2HNO_3 + 3H_2SO_4 = 3Fe_2(SO_4)_3 + 4H_2O + 2NO$$

$$10FeSO_4 + K_2O . 2MnO . O_5 + 8H_2SO_4 = 5Fe_2(SO_4)_3 + K_2SO_4 + 2MnSO_4 + 8H_2O$$

$$6FeSO_4 + K_2O . Cr_2O_3 . O_3 + 7H_2SO_4 = 3Fe_2(SO_4)_3 + Cr_2(SO_4)_3 + K_2SO_4 + 7H_2O.$$

Potassium sulphocyanate, no colouration with *pure* ferrous salts.

Caustic potash, same result as ammonia.

Ammonium sulphide, black ppt. of ferrous sulphide FeS.

Ferric Salts.

Solutions of ferric salts (*e.g.* Fe_2Cl_6) give with

Ammonia, reddish-brown ppt. of hydrated ferric oxide,

$$Fe_2Cl_6 + 6NH_3 + 5H_2O = Fe_2O_3 . 2H_2O + 6NH_4Cl.$$

Potassium ferrocyanide, dark blue ppt. of "Prussian blue." Usually represented †

$$2Fe_2Cl_6 + 3K_4(FeCy_6) = Fe'''_4(FeCy_6)^{iv}_3 + 12KCl.$$

Potassium sulphocyanate, blood-red colouration, due to ferric sulphocyanate, $Fe_2(CyS)_6$. Bleached by mercuric chloride.

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* Perhaps $2FeSO_4 + K_6(Fe_2Cy_{12}) = Fe''_2K_2(Fe_2Cy_{12}) + 2K_2SO_4$.

† But the ppt. always contains potassium. If excess of ferrocyanide be employed, the reaction appears to be $Fe'''_2Cl_6 + 2K_4(FeCy_6)^{iv} = K_2Fe''_2(Fe_2Cy_{12})^{vi} + 6KCl$.

Ferric Salts (*continued*).

Sodium phosphate, yellowish-white ppt. of ferric phosphate,

$$Fe_2Cl_6 + 2\,Na_2HPO_4 = 2FePO_4 + 4NaCl + 2HCl.$$

This ppt. is soluble in HCl, but insoluble in acetic acid. If an acid phosphate be used, as above, the precipitation is incomplete unless the free acid be neutralized, or replaced by acetic acid.

Potassium ferricyanide, no ppt. (brown or green colouration).

Ammonium sulphide, black ppt. of ferr*ous* sulphide, free sulphur separating.

Caustic potash, same ppt. as ammonia.

Reducing agents such as H_2S, SO_2, $SnCl_2$, Zn, &c., convert ferr*ic* into ferr*ous* compounds.

Compounds of iron heated in the borax bead, give, in the oxidizing flame, an orange yellow while hot, pale yellow when cold. In reducing flame, bottle-green bead, especially with ferrous salts.

CHROMIUM.

Any chromium compound fused with Na_2CO_3 on platinum foil in presence of air (or with addition of a little KNO_3) gives a yellow mass of sodium chromate; *e.g.*

$$Cr_2O_3 + 2Na_2CO_3 + O_3 = 2Na_2CrO_4 + 2CO_2.$$

This may be dissolved in water acidified with acetic acid, and tested for chromate as below with lead acetate.

Before the blow-pipe, chromium compounds give a green borax bead in both flames.

Chromic Salts.

Solutions of chromic salts (*e.g.* Cr_2Cl_6) give with

Ammonia, bluish-green ppt. of chromic hydroxide,

$$Cr_2Cl_6 + 6NH_3 + 6H_2O = Cr_2(OH)_6 + 6NH_4Cl.$$

This ppt. is partly soluble in excess of ammonia, giving a pink solution, but is completely reprecipitated by boiling for some time.

Caustic potash gives same result as ammonia, easily soluble in excess, forming a green solution. Reprecipitated by continued boiling.

Many *oxidising agents*, *e.g.* NaClO, PbO_2, $K_2Mn_2O_8$, (in presence of caustic potash), convert chromic salts into chromates.

Ammonium sulphide, bluish-green ppt. of chromic hydroxide $Cr_2(OH)_6$, with evolution of H_2S.

Sodium phosphate, green ppt. of chromic phosphate $CrPO_4$, sparingly soluble in acetic acid.

Chromates.

Solutions of chromates (*e.g.* K_2CrO_4) give with

Lead acetate, yellow ppt. of lead chromate, insoluble in acetic acid,

$$K_2CrO_4 + (CH_3CO_2)_2Pb = PbCrO_4 + 2CH_3CO_2K.$$

Hydrosulphuric acid, in presence of HCl, gives a green solution of chromic chloride, and a white ppt. of sulphur,

$$2K_2CrO_4 + 10HCl + 3H_2S = Cr_2Cl_6 + 4KCl + 8H_2O + 3S.$$

Many other reducing agents, *e.g.* stannous chloride, sulphur dioxide, alcohol, ferrous salts, likewise give green solutions of chromic salts.

$$\begin{aligned}
{}^{*}(K_2O)_2 . Cr_2O_3 . O_3 + 3SnCl_2 + 16HCl &= Cr_2Cl_6 + 3SnCl_4 + 4KCl + 8H_2O \\
(K_2O)_2 . Cr_2O_3 . O_3 + 3SO_2 + 2H_2SO_4 &= Cr_2(SO_4)_3 + 2K_2SO_4 + 2H_2O \\
(K_2O)_2 . Cr_2O_3 . O_3 + 3C_2H_5OH + 5H_2SO_4 &= Cr_2(SO_4)_3 + 3CH_3COH + 2K_2SO_4 + 8H_2O \\
(K_2O)_2 . Cr_2O_3 . O_3 + 6FeSO_4 + 8H_2SO_4 &= Cr_2(SO_4)_3 + 3Fe_2(SO_4)_3 + 2K_2SO_4 + 8H_2O.
\end{aligned}$$

Peroxide of Hydrogen, added to a *dilute* solution, acidified with dilute H_2SO_4 and shaken up with ether, gives a deep blue solution (due to perchromic acid $H_2Cr_2O_8$(?))

Silver nitrate, crimson ppt. of silver chromate Ag_2CrO_4, soluble in dilute HNO_3.

Barium chloride, yellow ppt. of barium chromate, insoluble in acetic acid.

Dry chromates, heated with concentrated H_2SO_4 and *sodium chloride*, give off red vapours of chromyl chloride CrO_2Cl_2.

Mercurous Nitrate, brick red ppt. of basic mercurous chromate $2Hg_2O . CrO_3$, which, on heating, gives off Hg and O, leaving Cr_2O_3.

ALUMINIUM.

Solutions of aluminium compounds, *e.g.* $Al_2(SO_4)_3$, give with

Ammonia, white gelatinous ppt. of aluminium hydroxide (or basic salt),

$$Al_2(SO_4)_3 + 6NH_3 + 6H_2O = Al_2(OH)_6 + 3(NH_4)_2SO_4.$$

This ppt. is somewhat soluble in excess, unless NH_4Cl is present, and the solution boiled.

Caustic potash, same ppt. as ammonia, easily soluble in excess, forming potassium aluminate, $3K_2O . Al_2O_3$; reprecipitated by *ammonium chloride*,

$$3K_2O . Al_2O_3 + 6NH_4Cl = Al_2O_3 . 3H_2O + 6KCl + 6NH_3.$$

Ammonium sulphide, white ppt. of aluminium hydroxide $Al_2(OH)_6$ (often discoloured).

Sodium phosphate, white ppt. of aluminium phosphate $AlPO_4$, insoluble in acetic acid.

Many solid aluminium compounds when heated on charcoal leave a white infusible mass, which, when moistened with cobalt nitrate and again heated, gives a sky-blue colour.

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* $(K_2O)_2 . Cr_2O_3 . O_3 = 2K_2CrO_4$.

ZINC.

Solutions of zinc salts (*e.g.* $ZnSO_4$) give with

Ammonium sulphide, white ppt. of zinc sulphide (often discoloured),

$$ZnSO_4 + (NH_4)_2S = ZnS + (NH_4)_2SO_4.$$

Soluble in dilute HCl, forming zinc chloride $ZnCl_2$. Insoluble in acetic acid. (Distinction from Mn.)

Caustic potash, white ppt. of zinc hydroxide,

$$ZnSO_4 + 2KHO = Zn(OH)_2 + K_2SO_4,$$

easily soluble in excess of *cold* caustic potash, forming potassium zincate $K_2O \cdot ZnO$, from which H_2S precipitates white zinc sulphide ZnS. On boiling the solution of $K_2O \cdot ZnO$, $Zn(OH)_2$ is reprecipitated, especially if dilute.

Ammonia, same ppt. as caustic potash, soluble in excess, and in ammonium chloride.

Hydrosulphuric acid, white ppt. of ZnS in *alkaline* solution, or one containing only *acetic* acid. No ppt. in presence of sufficient free HCl, H_2SO_4, or HNO_3.

Potassium ferrocyanide, white ppt. of zinc ferrocyanide, insoluble in dilute HCl.

Potassium ferricyanide, orange ppt. soluble in HCl.

Solid zinc compounds heated on charcoal before the blow-pipe with Na_2CO_3 give a residue which is yellow while hot, white when cold. This residue, when moistened with cobalt nitrate and again heated, gives a bright green mass.

MANGANESE.

General Reactions.

Any compound of manganese when fused in platinum foil with Na_2CO_3 in presence of air (or with addition of a little KNO_3) give a blue-green mass of sodium manganate, *e.g.*

$$MnSO_4 + 2Na_2CO_3 + O_2 = Na_2MnO_4 + Na_2SO_4 + 2CO_2.$$

All compounds of manganese after boiling with HCl, give the reactions of mangan*ous* salts.

Traces of manganese compounds (in absence of Cl and Br) when boiled with nitric acid and peroxide of lead, give a pink colour, due to permanganic acid $H_2Mn_2O_8$.

In the borax bead manganese compounds give a violet colour in the oxidizing flame, colourless in reducing flame.

Manganous Salts.

Solutions of manganous salts (*e.g.* $MnCl_2$) give with

Ammonium sulphide, buff or pinkish ppt. of manganous sulphide,

$$MnCl_2 + (NH_4)_2S = MnS + 2NH_4Cl.$$

This ppt. dissolves in dilute HCl, forming manganous chloride $MnCl_2$. Soluble also in acetic acid. Not precipitated in presence of large excess of ammonia.

Caustic potash, white ppt. of manganous hydroxide, insoluble in excess,

$$MnCl_2 + 2KHO = Mn(OH)_2 + 2KCl.$$

This ppt. rapidly absorbs oxygen from the air, forming brown hydrated sesquioxide $Mn_2O_3 . H_2O$.

Ammonia, same ppt. as caustic potash. No ppt., however, in presence of ammonium chloride, which forms the soluble double chloride $MnCl_2 . 2NH_4Cl$. This solution readily absorbs oxygen from the air, brown $Mn_2O_3 . H_2O$ being precipitated.

Sodium hypochlorite, dark brown ppt. of hydrated manganese dioxide $MnO_2 . H_2O$.

Hydrosulphuric acid gives no ppt. in presence of a free acid, even acetic. In alkaline solution MnS is precipitated.

Manganic Salts.

These are very unstable, and usually decompose when dissolved in water. They form red or violet solutions in acids.

Heated with *Hydrochloric acid* they evolve chlorine, *e.g.*

$$Mn_2(SO_4)_3 + 2HCl = 2MnSO_4 + H_2SO_4 + Cl_2.$$

Caustic potash gives a dark brown ppt. of hydrated manganese sesquioxide,

$$Mn_2(SO_4)_3 + 6KHO = Mn_2O_3 . H_2O + 3K_2SO_4 + 2H_2O.$$

Manganates and Permanganates.

Solutions of manganates (*e.g.* $K_2MnO_4 = K_2O . MnO_3$) are green; those of permanganates (*e.g.* $K_2Mn_2O_8 = K_2O . Mn_2O_7$) rose or purple.

Manganates in solution are very unstable, except in presence of free alkali. In neutral solution they decompose as follows:

$$3\underbrace{(K_2O . MnO_3)}_{\text{green solution}} + 3H_2O = \underbrace{K_2O . Mn_2O_7}_{\text{pink solution}} + \underbrace{MnO_2 . H_2O}_{\text{brown ppt.}} + 4KHO.$$

Chlorine converts manganates into permanganates,

$$2K_2MnO_4 + Cl_2 = K_2Mn_2O_8 + 2KCl.$$

Permanganates boiled with alkalis, become manganates, oxygen being evolved,

$$2K_2Mn_2O_8 + 4KHO = 4K_2MnO_4 + 2H_2O + O_2.$$

Permanganates (or manganates) when heated with strong *sulphuric acid* evolve oxygen,

$$2(K_2O . 2MnO . O_5) + 6H_2SO_4 = 2K_2SO_4 + 4MnSO_4 + 6H_2O + 5O_2;$$

and with strong *hydrochloric acid* evolve chlorine,

$$K_2O . 2MnO . O_5 + 16HCl = 2KCl + 2MnCl_2 + 8H_2O + 5Cl_2.$$

In presence of free acid, they convert—

Ferrous salts into ferric salts,

$$10Fe''SO_4 + 5H_2SO_4 + 5O = 5Fe'''_2(SO_4)_3 + 5H_2O.$$

Stannous salts into stannic salts,

$$5SnCl_2 + 5Cl_2 = 5SnCl_4.$$

Oxalic acid into carbon dioxide and water,

$$5\begin{cases} COOH \\ COOH \end{cases} + 5O = 10CO_2 + 5H_2O.$$

Nitrous acid into nitric acid,

$$5HNO_2 + 5O = 5HNO_3.$$

Sulphurous acid into sulphuric acid,

$$5H_2SO_3 + 5O = 5H_2SO_4.$$

NICKEL.

Solutions of nickel salts (*e.g.* $NiSO_4$) give with

Ammonium sulphide, black ppt.,

$$NiSO_4 + (NH_4)_2S = NiS + (NH_4)_2SO_4.$$

This ppt. is nearly insoluble in dilute HCl. Soluble in aqua regia, forming nickel chloride $NiCl_2$. Somewhat soluble in excess of ammonium sulphide (especially yellow), forming a dark-coloured solution. [It may be reprecipitated from this solution by adding ammonium acetate and boiling, or by acidifying with acetic acid.]

Caustic potash, pale green ppt. of nickelous hydroxide, insoluble in excess, soluble in ammonium salts,

$$NiSO_4 + 2KHO = Ni(OH)_2 + K_2SO_4.$$

Potassium cyanide (pure), greenish-yellow ppt. of nickelous cyanide,

$$NiSO_4 + 2KCy = NiCy_2 + K_2SO_4.$$

This ppt. redissolves easily in excess of potassium cyanide, forming a double cyanide $NiCy_2 . 2KCy$, which is not altered by boiling with excess of KCy in presence of air.

This double cyanide gives a pale green ppt. with mercuric oxycyanide*, and a black ppt. with sodium hypochlorite on gently warming, thus

$$NiCy_2 + HgO . HgCy_2 + H_2O = \underbrace{Ni(OH)_2}_{\text{greenish ppt.}} + 2HgCy_2$$

$$2NiCy_2 + NaClO + 5H_2O = \underbrace{Ni_2(OH)_6}_{\text{black ppt.}} + NaCl + 4HCy.$$

Potassium nitrite, in presence of acetic acid no ppt.

Ammonia, greenish ppt. of nickelous hydroxide, soluble in excess, forming a blue solution. Soluble in ammonium salts.

Hydrosulphuric acid, no ppt. in presence of free HCl.

Potassium ferricyanide, yellowish-brown ppt. of nickel ferricyanide, soluble in *ammonia*.

Nickel compounds give, in the borax bead, a brownish-violet colour in the oxidizing flame, often grey in the reducing flame.

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* Prepared by boiling mercuric oxide (freshly pptd.) with solution of mercuric cyanide and filtering.

COBALT.

Solutions of cobaltous salts, *e.g.* $Co(NO_3)_2$, give with

Ammonium sulphide, black ppt. of cobalt sulphide,

$$Co(NO_3)_2 + (NH_4)_2S = CoS + 2NH_4NO_3.$$

This ppt. is almost insoluble in dilute HCl. Soluble in aqua regia, forming cobaltous chloride $CoCl_2$.

Caustic potash, blue ppt. of basic salt, insoluble in excess, becoming green or grey on exposure to the air, owing to formation of sesquioxide. The blue ppt. turns to red cobaltous hydroxide $Co(OH)_2$ on heating.

Potassium cyanide (pure), brown ppt. of cobaltous cyanide,

$$Co(NO_3)_2 + 2KCy = CoCy_2 + 2KNO_3.$$

This ppt. redissolves easily in excess of potassium cyanide, forming a double cyanide $CoCy_2 . 2KCy$*. This solution, if boiled (with slight excess of KCy) in presence of air, is converted into potassium cobalticyanide†,

$$2\,(CoCy_2 2KCy) + 4KCy + H_2O + O = K_6Co_2Cy_{12} + 2KHO.$$

This compound is not precipitated by sodium hypochlorite (at gentle heat), or by mercuric oxycyanide.

Potassium nitrite (concentrated solution) in presence of acetic acid, yellow crystalline ppt. after a time, probably $K_6Co_2(NO_2)_{12} . 3H_2O$.

Ammonia in small quantity gives same result as caustic potash. More ammonia dissolves it, forming a brownish-red liquid which absorbs oxygen from the air becoming red.

Potassium ferricyanide, reddish-brown ppt. of cobalt ferricyanide, insoluble in *ammonia*.

Potassium sulphocyanate in *strong* solutions, blue colour, soluble in ether.

Cobalt compounds give in the borax bead a blue colour in both flames.

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* Derived from 4HCy by replacement of H_4 by Co'' and K'_2.

† Derived from $H_6Co_2Cy_{12}$ by replacement of H_6 by K_6.

BARIUM.

Solutions of barium salts (*e.g.* $BaCl_2$) give with

Ammonium carbonate, white ppt. of barium carbonate,

$$BaCl_2 + (NH_4)_2CO_3 = BaCO_3 + 2NH_4Cl,$$

soluble in *dilute* HCl or HNO_3.

Calcium sulphate, immediate white ppt. of barium sulphate,

$$BaCl_2 + CaSO_4 = BaSO_4 + CaCl_2.$$

Hydrofluosilicic acid, colourless crystalline ppt. of barium silicofluoride,

$$BaCl_2 + H_2SiF_6 = BaSiF_6 + 2HCl,$$

somewhat soluble in dilute acids, unless alcohol be added.

Potassium chromate, yellow ppt. of barium chromate, even in dilute solutions,

$$BaCl_2 + K_2CrO_4 = BaCrO_4 + 2KCl,$$

nearly insoluble in *acetic* acid. Soluble in HCl.

Barium salts are precipitated from their solutions by strong HCl or HNO_3. Barium chloride is insoluble in alcohol.

Dilute sulphuric acid, or any soluble sulphate, white ppt. of barium sulphate $BaSO_4$.

Sodium phosphate, white ppt. of barium phosphate, soluble in dil. HCl, HNO_3, and acetic acid.

Ammonium oxalate, white ppt. of barium oxalate $Ba(CO_2)_2$, soluble in HCl, very sparingly soluble in acetic acid.

Barium compounds moistened with HCl, and held in the edge of a Bunsen flame on platinum wire, give an apple-green colour.

CALCIUM.

Solutions of calcium salts (*e.g.* $CaCl_2$) give with

Ammonium carbonate, white ppt. of calcium carbonate,

$$CaCl_2 + (NH_4)_2CO_3 = CaCO_3 + 2NH_4Cl$$

Calcium sulphate, no ppt.

Hydrofluosilicic acid, no ppt.

Potassium chromate, no ppt.

Ammonium oxalate, white ppt. of calcium oxalate, even in very dilute solutions,

$$CaCl_2 + \left\{\begin{matrix}CO_2NH_4\\CO_2NH_4\end{matrix}\right. = \left\{\begin{matrix}CO_2\\CO_2\end{matrix}\right.Ca + 2NH_4Cl.$$

This ppt. is insoluble in *acetic acid;* soluble in dilute HCl or HNO_3.

Calcium chloride and calcium nitrate are both soluble in absolute alcohol.

Sulphuric acid or *soluble sulphates*, white ppt. of calcium sulphate in strong solutions only. Readily soluble in strong (ammoniacal) solution of *ammonium sulphate*.

Sodium phosphate, white ppt. of calcium phosphate, soluble in HCl, HNO_3, and acetic acid.

Calcium compounds examined by the flame test give an orange-red colour.

STRONTIUM.

Solutions of strontium salts, *e.g.* $Sr(NO_3)_2$, give with

Ammonium carbonate, white ppt. of strontium carbonate,

$$Sr(NO_3)_2 + (NH_4)_2CO_3 = SrCO_3 + 2NH_4NO_3.$$

Calcium sulphate, white ppt. of strontium sulphate *after a time*, or on heating,

$$Sr(NO_3)_2 + CaSO_4 = SrSO_4 + Ca(NO_3)_2.$$

Hydrofluosilicic acid, no ppt.

Potassium chromate, no ppt. in dilute solutions.

Strontium chloride is soluble in absolute alcohol; strontium nitrate insoluble.

Dilute sulphuric acid or *soluble sulphates*, white ppt. of strontium sulphate insoluble in strong solution of *ammonium sulphate* (ammoniacal).

Ammonium oxalate, white ppt. of strontium oxalate $Sr(CO_2)_2$, sparingly soluble in acetic acid, soluble in HCl.

Sodium phosphate, white ppt. of strontium phosphate, soluble in HCl, HNO_3, and acetic acid.

Strontium compounds examined by the flame test give a characteristic crimson colour.

MAGNESIUM.

Solutions of magnesium salts (*e.g.* $MgSO_4$) give with

Sodium phosphate, in presence of ammonia (and ammonium chloride), white ppt. of magnesium ammonium phosphate,

$$MgSO_4 + Na_2HPO_4 + NH_3 = MgNH_4PO_4 + Na_2SO_4.$$

This ppt. is crystalline from dilute solutions.

Ammonia, white ppt. of magnesium hydroxide, (in part)

$$MgSO_4 + 2NH_3 + 2H_2O = Mg(OH)_2 + (NH_4)_2SO_4,$$

but no ppt. in presence of sufficient ammonium chloride, since the soluble double chloride $MgCl_2 . 2NH_4Cl$ formed is not decomposed by ammonia.

Ammonium carbonate, white ppt. of magnesium carbonate, but no ppt. in presence of sufficient ammonium chloride.

Barium hydroxide, in absence of ammonium salts, white ppt. of magnesium hydroxide $Mg(OH)_2$.

Caustic potash, same ppt.

Some magnesium compounds (*e.g.* oxide, sulphate, carbonate) heated on charcoal, moistened with cobalt nitrate, and again heated, give a pale rose colour.

AMMONIUM.

Ammonium compounds (*e.g.* NH_4Cl), when boiled with caustic potash, evolve ammonia,

$$NH_4Cl + KHO = KCl + H_2O + NH_3.$$

The ammonia may be recognized by its odour, and action on red litmus.

Solutions of ammonium salts give with

Platinic chloride (in presence of HCl if necessary), yellow ppt. of ammonium platinic chloride,

$$2NH_4Cl + PtCl_4 = (NH_4)_2PtCl_6.$$

This precipitation is more complete in presence of alcohol.

Nessler's solution (potassium mercuric iodide with excess of caustic potash) gives, even in very dilute solutions, a brown or yellow colouration (or precipitate in stronger solutions) due to dimercuric-ammonium iodide,

$$NH_4Cl + 2(HgI_2 . 2KI) + 4KHO = NHg''_2I . H_2O + 7KI + KCl + 3H_2O.$$

Tartaric acid, white crystalline ppt. on shaking (in strong solutions) of ammonium hydrogen tartrate

$$\left\{\begin{array}{l}COONH_4\\CHOH\\CHOH\\COOH\end{array}\right.$$

Ammonium compounds heated before the blow-pipe volatilize entirely or in part.

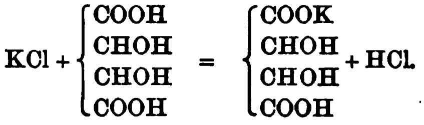
POTASSIUM.

Solutions of potassium salts (*e.g.* KCl), *if not too dilute*, give with

Platinic chloride (in presence of HCl if necessary), yellow crystalline ppt. of potassium platinic chloride, especially on stirring,

$$2KCl + PtCl_4 = K_2PtCl_6.$$

Tartaric acid, white crystalline ppt. on shaking, of potassium hydrogen tartrate,



$$\mathrm{KCl} + \left\{\begin{array}{l}\mathrm{COOH}\\ \mathrm{CHOH}\\ \mathrm{CHOH}\\ \mathrm{COOH}\end{array}\right. = \left\{\begin{array}{l}\mathrm{COOK}\\ \mathrm{CHOH}\\ \mathrm{CHOH}\\ \mathrm{COOH}\end{array}\right. + \mathrm{HCl}.$$

Hydrofluosilicic acid, white gelatinous ppt. of potassium silicofluoride K_2SiF_6.
Picric acid (in strong solutions), yellow crystalline ppt. of potassium picrate $C_6H_2(NO_2)_3 . OK$.

All of the above reactions of potassium are more complete in the presence of *alcohol*.

Potassium salts examined by the flame reaction, give a violet colour.

SODIUM.

Solutions of sodium compounds give no characteristic reaction with any of the common reagents, nearly all sodium salts being soluble.

Dioxytartaric acid, when neutralized with ammonia, gives a white precipitate of sodium dioxytartrate $Na_2C_4H_4O_8 . 2\frac{1}{2}H_2O$. A few crystals of the acid should be dissolved in a drop or two of water on a watch-glass, mixed with the solution to be tested, and ammonia added till neutral or alkaline. The precipitate appears in lines on stirring.

Potassium metantimoniate gives in neutral solutions a white ppt. of acid sodium metantimoniate, $Na_2O . H_2O . Sb_2O_5 . 6H_2O$.

This test is not often used. Ammonium, calcium, &c., give a similar reaction.

Sodium compounds, heated on platinum wire in a Bunsen flame, give a strong yellow colouration.

PRINCIPAL REACTIONS OF THE MORE COMMON ACID RADICLES.

SULPHATES.

Heated with strong H_2SO_4, no result.

Solutions of sulphates (*e.g.* K_2SO_4) give with

Barium chloride, white ppt. of barium sulphate, *insoluble* in HCl or HNO_3,

$$K_2SO_4 + BaCl_2 = BaSO_4 + 2KCl.$$

Strontium chloride (or nitrate), white ppt. of strontium sulphate, $SrSO_4$ (slowly in dilute solutions).

Calcium chloride, white ppt. of calcium sulphate $CaSO_4$, in strong solutions only; soluble in much water.

Lead acetate, white ppt. of lead sulphate $PbSO_4$, soluble in *ammonium acetate*.

Dry sulphates (like all sulphur compounds), when heated with sodium carbonate and charcoal in the reducing flame, give sodium sulphide, which when placed on a silver coin and moistened with water, gives a black stain of silver sulphide Ag_2S.

SULPHITES.

(*E.g.* Na_2SO_3.) Heated with HCl, sulphur dioxide is evolved,

$$Na_2SO_3 + 2HCl = 2NaCl + H_2O + SO_2.$$

The evolved gas may be recognized by its odour, and action on $K_2Cr_2O_7$.

$$\underset{\text{(orange)}}{K_2Cr_2O_7} + H_2SO_4 + 3SO_2 = K_2SO_4 + \underset{\text{(green)}}{Cr_2(SO_4)_3} + H_2O.$$

Treated with HCl and *metallic zinc*, hydrosulphuric acid is evolved (detected by lead acetate, &c.),

$$Zn + 2HCl = ZnCl_2 + H_2$$

and

$$SO_2 + 3H_2 = 2H_2O + H_2S.$$

Solutions of sulphites give with

Barium chloride, white ppt. of barium sulphite,

$$Na_2SO_3 + BaCl_2 = BaSO_3 + 2NaCl,$$

easily soluble in dilute HCl; from this solution oxidizing agents, *e.g.* chlorine water, precipitate white barium sulphate, insoluble in HCl,

$$BaSO_3 + H_2O + Cl_2 = BaSO_4 + 2HCl.$$

Silver nitrate, white ppt. of silver sulphite, Ag_2SO_3, soluble in dilute HNO_3. This ppt. darkens when heated, metallic silver and sulphuric acid being formed,

$$Ag_2SO_3 + H_2O = 2Ag + H_2SO_4.$$

Free iodine is decolourized, thus:

$$Na_2SO_3 + H_2O + I_2 = Na_2SO_4 + 2HI.$$

THIOSULPHATES.

Formerly called Hyposulphites.

(*E.g.* $Na_2S_2O_3$.) Treated with HCl, sulphur dioxide is evolved, and yellow sulphur separates, especially on heating,

$$Na_2S_2O_3 + 2HCl = 2NaCl + H_2O + SO_2 + S.$$

Treated with metallic zinc in excess and HCl, H_2S is evolved as with sulphites, especially on heating.

Thiosulphates (*continued*).

Solutions of thiosulphates give with

Lead acetate, white ppt. of lead thiosulphate,

$$Na_2S_2O_3 + (CH_3CO_2)_2Pb = PbS_2O_3 + 2CH_3COONa.$$

This ppt. on heating blackens, owing to formation of lead sulphide,

$$PbS_2O_3 + H_2O = PbS + H_2SO_4.$$

Silver and *mercurous* salts behave similarly.

Free iodine is at once decolourized,

$$2Na_2S_2O_3 + I_2 = Na_2S_4O_6 + 2NaI.$$

Ferric chloride, transient violet colouration (afterwards reduced to ferrous chloride).

SULPHIDES.

Heated alone in a current of air (*e.g.* in an open tube) sulphur dioxide is evolved.

Treated with HCl, many sulphides evolve hydrosulphuric acid. [Some require boiling with strong acid; others (*e.g.* FeS_2) are not decomposed unless zinc be added.]

$$FeS + 2HCl = FeCl_2 + H_2S.$$

The evolved gas is recognized by its odour, action on lead acetate, &c.

Soluble sulphides, *e.g.* $(NH_4)_2S$, give with

Lead acetate, black ppt. of lead sulphide,

$$(NH_4)_2S + (CH_3CO_2)_2Pb = PbS + 2CH_3COO(NH_4).$$

Sodium nitroprusside, $Na_4Fe_2(NO)_2Cy_{10}$, gives a fine purple colouration, probably due to a double salt of the sulphide and nitroprusside. Free H_2S does not give the reaction.

Metallic silver is stained black owing to formation of silver sulphide Ag_2S.

SILICOFLUORIDES.

(*E.g.* K_2SiF_6) when heated in a platinum vessel with strong H_2SO_4, evolve silicon tetrafluoride and hydrofluoric acid gases,

$$K_2SiF_6 + H_2SO_4 = K_2SO_4 + SiF_4 + 2HF.$$

SiF_4 deposits white hydrated silica on a wet rod, and HF will etch glass. [See Fluorides.]

Solutions of silicofluorides, or hydrofluosilicic acid, give with

Barium chloride, colourless crystalline ppt. of barium silicofluoride,

$$H_2SiF_6 + BaCl_2 = BaSiF_6 + 2HCl.$$

This ppt. is nearly insoluble in dilute HCl.

Strontium chloride, no ppt.

Potassium chloride, gelatinous ppt. of potassium silicofluoride,

$$H_2SiF_6 + 2KCl = K_2SiF_6 + 2HCl.$$

FLUORIDES.

(*E.g.* CaF_2) Heated in a platinum vessel with concentrated H_2SO_4 evolve hydrofluoric acid,

$$CaF_2 + H_2SO_4 = CaSO_4 + 2HF.$$

The evolved gas will corrode glass, owing to its action on silica,

$$SiO_2 + 4HF = SiF_4 + 2H_2O.$$

Heated with concentrated H_2SO_4 in presence of *silica* or silicates, *e.g.* in a test-tube, silicon tetrafluoride is evolved:

$$2CaF_2 + 2H_2SO_4 + SiO_2 = 2CaSO_4 + 2H_2O + SiF_4.$$

A wet rod held in this gas becomes coated with hydrated silica,

$$3SiF_4 + 4H_2O = \underset{\text{white deposit}}{SiO_2 . 2H_2O} + 2H_2SiF_6.$$

Solutions of fluorides (*e.g.* NaF) give with

Calcium chloride, white gelatinous ppt. of calcium fluoride,

$$2NaF + CaCl_2 = CaF_2 + 2NaCl.$$

Sparingly soluble in HCl; nearly insoluble in *acetic* acid.

Barium chloride, white ppt. of barium fluoride BaF_2.

OXALATES.

$\left(E.g. \left\{\begin{matrix}\mathrm{COOK}\\ \mathrm{COOK}\end{matrix}\right.\right)$ Heated with strong H_2SO_4, carbon monoxide and carbon dioxide are evolved,

$$\left\{\begin{matrix}\mathrm{COOK}\\ \mathrm{COOK}\end{matrix}\right. + H_2SO_4 = K_2SO_4 + CO + CO_2 + H_2O.$$

The CO will burn with a blue flame, and the CO_2 may be detected by lime water. (See Carbonates.)

Treated with H_2SO_4 and manganese dioxide, carbon dioxide alone is evolved,

$$\left\{\begin{matrix}\mathrm{COOK}\\ \mathrm{COOK}\end{matrix}\right. + MnO_2 + 2H_2SO_4 = K_2SO_4 + MnSO_4 + 2H_2O + 2CO_2.$$

Solutions of oxalates give with

Calcium chloride, white ppt. of calcium oxalate,

$$\left\{\begin{matrix}\mathrm{COOK}\\ \mathrm{COOK}\end{matrix}\right. + CaCl_2 = \left\{\begin{matrix}\mathrm{COO}\\ \mathrm{COO}\end{matrix}\right. Ca + 2KCl,$$

soluble in HCl, but insoluble in *acetic* acid. This ppt. is produced even in very dilute solutions.

Barium chloride and *Strontium chloride*, white ppts. of barium oxalate $(CO_2)_2Ba$ and strontium oxalate $(CO_2)_2Sr$ respectively.

Silver nitrate, white ppt. of silver oxalate $\left\{\begin{matrix}\mathrm{COOAg}\\ \mathrm{COOAg}\end{matrix}\right.$, soluble in dilute HNO_3.

PHOSPHATES.

Orthophosphates.

Heated with strong H_2SO_4, no result.

Solutions of orthophosphates (*e.g.* Na_2HPO_4) give with

Calcium chloride, white ppt. of calcium hydrogen phosphate,

$$Na_2HPO_4 + CaCl_2 = CaHPO_4 + 2NaCl,$$

soluble in HCl, HNO_3, and acetic acid.

[If ammonia is added, $Ca_3(PO_4)_2$ is precipitated instead of $CaHPO_4$.]

Orthophosphates (*continued*).

Ferric chloride, yellowish-white ppt. of ferric phosphate,

$$2Na_2HPO_4 + Fe_2Cl_6 = 2Fe(PO_4) + 4NaCl + 2HCl,$$

soluble in HCl or HNO_3, insoluble in *acetic* acid. [This precipitation, in case of acid phosphates as above, is therefore incomplete, unless the HCl be neutralized or replaced by acetic acid.]

Magnesium sulphate, in presence of ammonia and ammonium chloride (the latter, to prevent precipitation of $Mg(OH)_2$ by the ammonia), white ppt. (crystalline in dilute solutions or on standing) of magnesium ammonium phosphate,

$$Na_2HPO_4 + MgSO_4 + NH_3 = Mg(NH_4)PO_4 + Na_2SO_4.$$

Silver nitrate, yellow ppt. of silver phosphate,

$$Na_2HPO_4 + 3AgNO_3 = Ag_3PO_4 + 2NaNO_3 + HNO_3,$$

soluble in *acids* and in *ammonia*. [The above precipitation is therefore incomplete unless the free HNO_3 be neutralized.]

Ammonium molybdate, $(NH_4)_2MoO_4$ in presence of strong HNO_3, on heating, canary yellow ppt. of ammonium phosphomolybdate. [Composition uncertain, approximately

$$2(NH_4)_3PO_4 . 22MoO_3 . 12H_2O.]$$

The $(NH_4)_2MoO_4$ must be added considerably in excess of the phosphate taken.

Free orthophosphoric acid does not coagulate albumin.

Barium, strontium, lead, aluminium, bismuth, &c. salts give white ppts. of their respective normal or acid phosphates.

Metaphosphates.

Solutions of metaphosphates (*e.g.* $NaPO_3$) give with

Silver nitrate, white ppt. of silver metaphosphate $AgPO_3$.

Magnesium sulphate, in presence of NH_3 and NH_4Cl, no ppt.

The free acid (or a metaphosphate + acetic acid) coagulates albumin.

Pyrophosphates.

Solutions of pyrophosphates (*e.g.* $Na_4P_2O_7$) give with

Silver nitrate, white ppt. of silver pyrophosphate $Ag_4P_2O_7$.

Magnesium sulphate, white ppt. of magnesium pyrophosphate $Mg_2P_2O_7$, soluble in excess.

The free acid does not coagulate albumin.

Boiled with water, in presence of acids, both meta- and pyro-phosphates are converted into orthophosphates.

ARSENATES.

Solutions of arsenates (*e.g.* K_3AsO_4) give the same reactions as phosphates with Ferric chloride, Magnesium sulphate, and Ammonium molybdate. Silver nitrate however gives, in neutral solution, a brick-red ppt. instead of yellow.

Arsenates are also distinguished (and separated) from phosphates by *hydrosulphuric acid* in presence of HCl, which gives first a white ppt. of sulphur, and afterwards a yellow ppt. of arsenious sulphide, on heating. [See Arsenic.]

BORATES.

(*E.g.* $Na_2B_4O_7$.) Mixed with strong H_2SO_4 and alcohol, the latter on being kindled burns with a green-edged flame.

Solutions of borates *acidified* with dilute HCl, colour turmeric paper orange or brown (in dilute solutions only on drying). This colour is changed to green or blackish by treatment with KHO.

Solutions of borates give with *calcium chloride*, *barium chloride*, or *silver nitrate*, white ppts. (such as $2CaO \,.\, H_2O \,.\, 3B_2O_3$, $BaBO_2$, $AgBO_2$, but variable in composition according to relative proportions used, temperature, &c.), readily soluble in dilute acids.

Strong Hydrochloric acid gives white crystalline ppt. of boric acid, in concentrated solutions,

$$Na_2O \,.\, 2B_2O_3 + 2HCl + 5H_2O = 2(B_2O_3 \,.\, 3H_2O) + 2NaCl.$$

SILICATES.

Insoluble in water, except those of K and Na.

Silica, or insoluble silicates, when fused with excess of K_2CO_3 or Na_2CO_3 in a platinum vessel, give silicates which are soluble in water, *e.g.*

$$SiO_2 + K_2CO_3 = SiO_2 \,.\, K_2O + CO_2.$$

These, when dissolved in water and poured into an excess of HCl, give a solution containing silicic acid,

$$SiO_2K_2O + 2HCl + H_2O = SiO_2 \,.\, 2H_2O + 2KCl.$$

A solution of silicic acid, or a solution of any soluble silicate in HCl, when evaporated to dryness, leaves a residue of SiO_2, which is insoluble in all acids except HF.

Silica or silicates treated with a fluoride (*e.g.* NH_4F, or CaF_2) and concentrated H_2SO_4 give off gaseous silicon tetrafluoride,

$$SiO_2 + 2H_2SO_4 + 4NH_4F = 2(NH_4)_2SO_4 + SiF_4 + 2H_2O.$$

Solutions of silicates (*e.g.* K_2SiO_3) give white ppt. with calcium chloride, barium chloride, or silver nitrate, of variable composition; easily decomposed by acids.

CARBONATES.

(*E.g.* Na_2CO_3) Treated with acids (either dilute or strong) CO_2 is evolved,

$$Na_2CO_3 + 2HCl = 2NaCl + H_2O + CO_2.$$

This gas, when passed into lime water, gives white ppt. of calcium carbonate,

$$CO_2 + Ca(HO)_2 = CaCO_3 + H_2O.$$

Excess of CO_2 redissolves the ppt.

$$CaCO_3 + H_2O + CO_2 = CaH_2(CO_3)_2.$$

Solutions of carbonates give with

Calcium chloride, white ppt. of calcium carbonate,

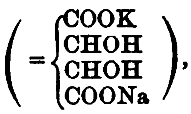
$$Na_2CO_3 + CaCl_2 = CaCO_3 + 2NaCl,$$

easily soluble in acids with effervescence.

Salts of almost all other metals give precipitates of carbonates or basic carbonates.

TARTRATES.

Solid tartrates heated with concentrated H_2SO_4 blacken immediately, owing to separation of carbonaceous matter, and evolve CO and CO_2.

Solutions of neutral tartrates, *e.g.* $KNa\bar{T}$ 

$$\left(= \begin{cases} COOK \\ CHOH \\ CHOH \\ COONa \end{cases} \right),$$

give with

Calcium chloride, white gelatinous ppt. of calcium tartrate,

$$KNa\bar{T} + CaCl_2 = Ca\bar{T} + KCl + NaCl,$$

soluble in HCl, HNO_3, and acetic acid*. Soluble also in cold KHO (after washing), repptd. by boiling. Neutral cupric chloride converts it into *insoluble* cupric tartrate.

Potassium acetate, in presence of *acetic acid*, white crystalline ppt. of potassium hydrogen tartrate, especially on shaking,

$$Na_2T + CH_3COOK + CH_3COOH = KH\bar{T} + 2CH_3COONa,$$

more complete in presence of alcohol. Soluble in KHO, forming normal tartrate $K_2\bar{T}$.

Silver nitrate, white ppt. of silver tartrate,

$$KNa\bar{T} + 2AgNO_3 = Ag_2\bar{T} + KNO_3 + NaNO_3,$$

soluble in HNO_3 and in NH_3.

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* After standing some time calcium tartrate usually becomes crystalline, and will not then dissolve in acetic acid.

Tartrates (*continued*).

If dissolved in the least possible quantity of NH_3, the solution on heating deposits a bright mirror of metallic silver on the side of the tube*.

Potassium permanganate (one or two drops), in presence of caustic potash, is reduced on heating, first to green manganate,

$$2(K_2O . Mn_2O_7) + 4KHO = 4(K_2O . MnO_3) + 2H_2O + O_2,$$

which is rapidly changed to a brown ppt. of hydrated manganese dioxide,

$$2(K_2OMnO_3) + 4H_2O = 2(MnO_2 . H_2O) + 4KHO + O_2,$$

the tartrate being oxidized to formate carbonate, &c.

If to a neutral or acid solution of a tartrate (or tartaric acid), a little *ferrous sulphate* be added, followed by a few drops of *hydrogen dioxide*, and lastly excess of *caustic potash*, a deep violet or blue colour is obtained. (Metals other than K, Na, and NH_4, must be absent.)

CITRATES.

Solid citrates treated with concentrated H_2SO_4, evolve CO, and on heating blacken slowly, evolving CO_2, acetone, &c.

Solutions of normal citrates, *e.g.* $Na_3\overline{Ci}$ ($= C_3H_4(OH)(COONa)_3$), give with

Calcium chloride, white ppt. of calcium citrate,

$$2Na_3\overline{Ci} + 3CaCl_2 = Ca_3\overline{Ci}_2 + 6NaCl,$$

soluble in dilute acids, and in *neutral cupric chloride*. Insoluble in cold KHO.

N.B. Normal ammonium citrate gives no ppt. till boiled.

Barium acetate, white ppt. of barium citrate $Ba_3\overline{Ci}_2$, especially in presence of alcohol and on long standing. (Tartaric acid must be first removed if present.)

Potassium permanganate, in presence of excess of KHO, is reduced on heating to green manganate, but is *not* further reduced, except after continued boiling.

Lime water added to *free* citric acid gives no ppt. (or only slight turbidity) in the cold, but on boiling, white calcium citrate is pptd.

Potassium acetate, no ppt. $AgNO_3$, white ppt. of $Ag_3\overline{Ci}$, which does not behave as with tartrate on dissolving in NH_3 and heating.

$FeSO_4$, H_2O_2, and KHO, as above, gives only a yellow or brown colour.

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* The safest method of trying the "mirror" test is as follows: 1st, add *dilute* NH_3 to $AgNO_3$ till the ppt. first formed is *nearly* redissolved. 2nd, make the solution to be tested neutral or faintly alkaline with KHO. Mix the two solutions and heat gently.

CHLORIDES.

(*E.g.* NaCl.) Solid chlorides treated with concentrated H_2SO_4, evolve ***hydrochloric acid*** in the cold,

$$NaCl + H_2SO_4 = NaHSO_4 + HCl,$$

colourless gas forming dense white fumes of NH_4Cl on coming in contact with NH_3.

Heated with ***manganese dioxide*** and strong H_2SO_4, ***chlorine*** is evolved.

$$2NaCl + 2H_2SO_4 + MnO_2 = Na_2SO_4 + MnSO_4 + 2H_2O + Cl_2,$$

yellowish-green gas, bleaches litmus, liberates I from KI.

Distilled with dry ***potassium dichromate*** and concentrated H_2SO_4, chromyl dichloride ("chlorochromic acid") is obtained,

$$4NaCl + K_2Cr_2O_7 + 3H_2SO_4 = K_2SO_4 + 2Na_2SO_4 + 2CrO_2Cl_2 + 3H_2O,$$

orange red gas condensing to a deep red liquid; decomposed on coming in contact with water, giving chromic and hydrochloric acids,

$$CrO_2Cl_2 + 2H_2O = H_2CrO_4 + 2HCl.$$

Solutions of chlorides give with

Silver nitrate, white ppt. of silver chloride which coagulates on shaking.

$$NaCl + AgNO_3 = AgCl + NaNO_3,$$

insoluble in HNO_3, soluble in NH_3. Turns violet on exposure to sunlight. Not decomposed on heating.

Lead acetate, white ppt. of lead chloride $PbCl_2$.

Mercurous nitrate, white ppt. of mercurous chloride Hg_2Cl_2.

HYPOCHLORITES.

(*E.g.* NaClO.) Treated with HCl, chlorine is evolved in the cold,

$$NaClO + HCl = NaCl + HClO,$$

and

$$HClO + HCl = H_2O + Cl_2.$$

Solutions of hypochlorites bleach litmus, indigo, ink, &c. They give with

Manganous chloride, brown ppt. of hydrated manganese dioxide,

$$NaClO + MnCl_2 + 2NaOH = MnO_2 . H_2O + 3NaCl.$$

Metallic mercury, on shaking, yellow deposit of mercuric oxide,

$$NaClO + Hg = NaCl + HgO.$$

Free hypochlorous acid shaken with metallic mercury gives yellow mercuric oxychloride, $HgCl_2 . HgO$; whereas free chlorine gives white mercurous chloride, Hg_2Cl_2.

CHLORATES.

(*E.g.* $KClO_3$.) Treated with concentrated H_2SO_4, the liquid turns yellow, and on heating, chlorine peroxide (yellow explosive gas) is evolved,

$$3KClO_3 + 2H_2SO_4 = KClO_4 + 2KHSO_4 + H_2O + 2ClO_2.$$

Heated with strong HCl, chlorine peroxide and chlorine are evolved,

$$2KClO_3 + 4HCl = 2KCl + 2H_2O + 2ClO_2 + Cl_2.$$

Most chlorates, when heated alone to redness, evolve oxygen and leave a chloride,

$$2KClO_3 = KClO_4 + KCl + O_2,$$

and at higher temperature, $KClO_4 = KCl + 2O_2$.

Solutions of chlorates acidulated with dilute H_2SO_4 bleach indigo only *on heating*. If a few drops of *sulphurous acid* be added, however, they do so *in the cold* (the chlorate being reduced to chlorite or hypochlorite).

IODIDES.

(*E.g.* KI.) Treated with concentrated H_2SO_4, iodine is liberated as a brown or black ppt. which, on heating, gives violet vapours,

$$2KI + 3H_2SO_4 = 2KHSO_4 + SO_2 + 2H_2O + I_2.$$

Free iodine colours starch-paste blue; the colour is temporarily destroyed by heat.

Solutions of iodides give with

Silver nitrate, pale yellow ppt. of silver iodide,

$$KI + AgNO_3 = AgI + KNO_3,$$

practically insoluble in ammonia. Insoluble in dilute HNO_3. Darkens on exposure to light.

Chlorine water, or *nitrous acid*, liberates iodine,

$$2KI + Cl_2 = 2KCl + I_2$$

$$2HI + 2HNO_2 = 2H_2O + 2NO + I_2.$$

If the mixture be shaken up with carbon disulphide, the iodine is dissolved out, giving a violet colour.

Cupric sulphate, dirty white ppt. of cuprous iodide, with liberation of iodine,

$$4KI + 2CuSO_4 = Cu_2I_2 + 2K_2SO_4 + I_2.$$

In presence of sulphurous acid no iodine is liberated,

$$2KI + 2CuSO_4 + H_2SO_3 + H_2O = Cu_2I_2 + 2KHSO_4 + H_2SO_4.$$

Lead acetate, yellow ppt. of lead iodide PbI_2, soluble in boiling water, crystallizing out on cooling.
Mercuric chloride, red ppt. of mercuric iodide HgI_2, soluble in KI and in $HgCl_2$.
Palladium nitrate, black ppt. of PdI_2.

IODATES.

(*E.g.* $K_2I_2O_6$.) Boiled with strong HCl, chlorine is evolved, together with iodine trichloride,

$$K_2I_2O_6 + 12HCl = 2KCl + 6H_2O + 2ICl_3 + 2Cl_2.$$

Strong H_2SO_4 liberates iodic acid, but no free iodine.

Solutions of iodates give with

Potassium iodide, in presence of a dilute acid (H_2SO_4, HCl, or CH_3COOH), liberation of iodine,

$$10HI + H_2I_2O_6 = 6H_2O + 6I_2.$$

Silver nitrate, white ppt. of silver iodate,

$$K_2I_2O_6 + 2AgNO_3 = Ag_2I_2O_6 + 2KNO_3,$$

sparingly soluble in cold dilute HNO_3. Easily soluble in ammonia, from which solution, SO_2 precipitates AgI, insoluble in ammonia.

Barium chloride, white crystalline ppt. of barium iodate $BaI_2O_6 . H_2O$.

Pyrogallol $C_6H_3(OH)_3$, brownish-red colour, sometimes ppt. of purpurogallin $C_{20}H_{16}O_9$.

BROMIDES.

(*E.g.* KBr.) Heated with concentrated H_2SO_4, bromine is evolved, mixed with hydrobromic acid,

$$KBr + H_2SO_4 = KHSO_4 + HBr$$

$$2HBr + H_2SO_4 = 2H_2O + SO_2 + Br_2.$$

Free bromine colours starch-paste yellow.

Heated with concentrated H_2SO_4 and manganese dioxide, bromine alone is evolved,

$$2KBr + 3H_2SO_4 + MnO_2 = 2KHSO_4 + MnSO_4 + 2H_2O + Br_2.$$

Solutions of bromides give with

Silver nitrate, yellowish-white ppt. of silver bromide,

$$KBr + AgNO_3 = AgBr + KNO_3,$$

sparingly soluble in ammonia. Insoluble in dilute HNO_3. Darkens on exposure to sunlight.

Chlorine water liberates free bromine,

$$2KBr + Cl_2 = 2KCl + Br_2.$$

If the mixture be shaken up with carbon disulphide, the bromine is dissolved out, giving a yellow or orange colour.

BROMATES.

(*E.g.* $KBrO_3$.) Heated with acids, bromic acid, $HBrO_3$, is liberated, which soon decomposes, giving off bromine and oxygen,

$$4HBrO_3 = 2H_2O + 2Br_2 + 5O_2.$$

Solutions of bromates give with

Silver nitrate, white ppt. of silver bromate,

$$KBrO_3 + AgNO_3 = AgBrO_3 + KNO_3,$$

sparingly soluble in dilute HNO_3. Soluble in ammonia.

Potassium bromide, in presence of cold dilute acid, liberation of bromine,

$$HBrO_3 + 5HBr = 3H_2O + 3Br_2.$$

CYANIDES.

Treated with dilute HCl, many cyanides, (*e.g.* KCy, or $ZnCy_2$) evolve hydrocyanic acid in the cold; others (*e.g.* $HgCy_2$, or AgCy) require strong HCl and heating.

$$KCy + HCl = KCl + HCy.$$

The evolved HCy may be absorbed by KHO (on a watch-glass or filter paper) and tested with iron salts as below; (or by $(NH_4)_2S_2$, and tested for *sulphocyanate* with HCl and Fe_2Cl_6—$(NH_4)_2S_2 + HCy = NH_4CyS + NH_4HS$).

Heated with excess of concentrated H_2SO_4, carbon monoxide is evolved (in part),

$$2KCN + 4H_2SO_4 + 2H_2O = 2KHSO_4 + 2NH_4HSO_4 + 2CO.$$

Solutions of cyanides (not mercuric cyanide) give with

Silver nitrate, white ppt. of silver cyanide,

$$KCy + AgNO_3 = AgCy + KNO_3,$$

insoluble in cold dilute HNO_3; easily soluble in ammonia, and in KCy. Not darkened by exposure to sunlight. On heating, it decomposes into metallic silver, cyanogen, and paracyanogen.

On adding successively *Ferrous sulphate*, *Ferric chloride*, and *hydrochloric acid*, a dark blue ppt., "Prussian blue," is obtained,

$$6KCy + FeSO_4 = K_4FeCy_6 + K_2SO_4$$

$$3K_4FeCy_6 + 2Fe_2Cl_6 = Fe_4(FeCy_6)_3{}^* + 12KCl.$$

(If the solution contains free acid to start with, KHO must be added before applying the test. The object of the HCl is to dissolve the hydroxides of iron which may have been precipitated by alkali present.)

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[*] See Ferric Salts.

CYANATES.

(*E.g.* KCNO.) Treated with dilute acids, carbon dioxide is evolved, and an ammonium salt formed, which may be identified as usual,

$$KCNO + HCl = KCl + HCNO$$

$$HCNO + H_2O + HCl = NH_4Cl + CO_2.$$

Traces of HCNO escape undecomposed, giving a pungent odour.

Solutions of cyanates treated with ammonium sulphate give ammonium cyanate, by double decomposition, which is soon transformed into urea,

$$NH_4CNO = CO(NH_2)_2.$$

The solution may be evaporated to dryness (on a water bath), the urea extracted with alcohol, and identified as usual.

Silver nitrate gives white ppt. of silver cyanate AgCNO, easily soluble in ammonia. Decomposed by acids as above.

FERROCYANIDES.

(*E.g.* $K_4FeC_6N_6 = K_4FeCy_6 + K_4FCy.$)

Heated with strong H_2SO_4, carbon monoxide is evolved

$$K_4FeC_6N_6 + 6H_2SO_4 + 6H_2O = 2K_2SO_4 + 3(NH_4)_2SO_4 + FeSO_4 + 6CO.$$

With dilute H_2SO_4, hydrocyanic acid is obtained,

$$2K_4FeCy_6 + 3H_2SO_4 = K_2Fe(FeCy_6) + 3K_2SO_4 + 6HCy.$$

Solutions of ferrocyanides give with

Silver nitrate, white ppt. of silver ferrocyanide,

$$K_4(FeCy_6) + 4AgNO_3 = Ag_4(FeCy_6) + 4KNO_3,$$

insoluble in dilute HNO_3 and in cold ammonia.

Ferric chloride, dark blue ppt. of "Prussian blue." (See Ferric salts.)

Ferrous sulphate, pale blue (white if quite pure) ppt. of potassium ferrous ferrocyanide,

$$K_4FeCy_6 + FeSO_4 = K_2Fe''FeCy_6 + K_2SO_4.$$

Cupric sulphate in excess, chocolate ppt. of cupric ferrocyanide,

$$K_4FeCy_6 + 2CuSO_4 = Cu_2(FeCy_6) + 2K_2SO_4.$$

Oxidizing agents, such as $K_2Mn_2O_8$ or chlorine, convert ferrocyanides into ferricyanides,

$$10K_4FeCy_6 + K_2O . 2MnO . O_5 + 8H_2SO_4 = 5K_6(Fe_2Cy_{12}) + 6K_2SO_4 + 2MnSO_4 + 8H_2O.$$

FERRICYANIDES.

(*E.g.* $K_6Fe_2C_{12}N_{12} = K_6Fe_2Cy_{12} = K_6(FdCy)_2$)

Heated with concentrated H_2SO_4, carbon monoxide is evolved (also CO_2, &c.) and ferric, potassium, and ammonium sulphates remain.

Solutions of ferricyanides give with

Silver nitrate, orange ppt. of silver ferricyanide,

$$K_6(Fe_2Cy_{12}) + 6AgNO_3 = Ag_6(Fe_2Cy_{12}) + 6KNO_3,$$

insoluble in dilute HNO_3, soluble in ammonia.

Ferrous sulphate, dark blue ppt. of ferrous ferricyanide (?) "Turnbull's blue."

$$K_6(Fe_2Cy_{12}) + 3FeSO_4 = Fe_3(Fe_2Cy_{12}) + 3K_2SO_4.$$

Potassium iodide (in presence of HCl) reduces ferri- to ferro-cyanides with liberation of iodine,

$$K_6Fe_2Cy_{12} + 2KI = 2K_4FeCy_6 + I_2.$$

Ferric chloride, no ppt. (brown solution, green if not quite pure).

Copper sulphate, yellowish-green ppt., *Nickel sulphate*, yellowish-green ppt. *Cobalt nitrate*, reddish-brown ppt. of the respective ferricyanides.

SULPHOCYANATES.

(*E.g.* KCNS.) Heated with strong HCl or H_2SO_4, carbon dioxide, hydrosulphuric acid, hydrocyanic acid, &c. are evolved,

$$KCNS + H_2SO_4 = KHSO_4 + HCNS$$

$$3HCNS = \underset{\text{(yellow ppt.)}}{H_2C_2N_2S_3} + HCN$$

$$HCNS + 2H_2O = CO_2 + H_2S + NH_3.$$

Solutions of sulphocyanates give with

Ferric chloride, blood-red colour due to ferric sulphocyanate $Fe_2(CNS)_6$. The colour is bleached by mercuric chloride, but not by dilute HCl.

Silver nitrate, white ppt. of silver sulphocyanate,

$$KCNS + AgNO_3 = AgCNS + KNO_3,$$

easily soluble in ammonia, insoluble in dilute HNO_3.

NITRATES.

(*E.g.* KNO_3.) Heated with concentrated H_2SO_4, nitric acid is evolved,

$$KNO_3 + H_2SO_4 = KHSO_4 + HNO_3.$$

Part of the HNO_3 usually splits up, giving yellow or brown vapours of lower nitrogen oxides, *e.g.*

$$4HNO_3 = 2H_2O + 4NO_2 + O_2.$$

Heated with concentrated H_2SO_4 and metallic copper, brownish-red fumes of nitrogen peroxide are given off,

$$2KNO_3 + 3H_2SO_4 + Cu = 2KHSO_4 + CuSO_4 + 2H_2O + 2NO_2.$$

Solutions of nitrates

Treated with excess of *ferrous sulphate* in the cold, and concentrated H_2SO_4 gradually poured in down the side of the vessel, give a brown ring where the liquids meet,

$$2KNO_3 + 6FeSO_4 + 4H_2SO_4 = K_2SO_4 + 3Fe_2(SO_4)_3 + 4H_2O + 2NO$$

and $$2FeSO_4 + NO = \underbrace{(2FeSO_4 . NO)}_{\text{brown compound}}.$$

Heated gently with amalgamated zinc they are reduced (in part at any rate) to nitrites,

$$KNO_3 + Zn = KNO_2 + ZnO,$$

which may be detected by the KI + HCl test, &c. [See Nitrites.]

Brucine in presence of concentrated H_2SO_4 gives a red colour.

Mixed with dilute H_2SO_4, solutions of nitrates do not bleach *indigo* in the cold (even in presence of SO_2), but do so on heating.

"*Nascent*" *hydrogen* (from Zn + KHO, &c.) converts them into ammonia. [See Nitrites.]

NITRITES.

(*E.g.* KNO_2.) Treated with dilute acids, nitric oxide is evolved,

$$KNO_2 + HCl = KCl + HNO_2$$

and $$3HNO_2 = HNO_3 + 2NO + H_2O.$$

The evolved gas unites with oxygen from the air, giving brownish-red fumes of N_2O_4.

Solutions of nitrites give with

Ferrous sulphate, even in neutral solution or in presence of *dilute* acid, a brown colour,

$$2KNO_2 + 2FeSO_4 + 2H_2SO_4 = K_2SO_4 + Fe_2(SO_4)_3 + 2H_2O + 2NO$$

and $$2FeSO_4 + NO = (2FeSO_4 . NO).$$

Nitrites (*continued*).

Potassium iodide, in presence of dilute HCl or H_2SO_4, liberation of iodine (detected by starch-paste or CS_2),

$$2KNO_2 + 2KI + 4HCl = 4KCl + 2H_2O + 2NO + I_2.$$

Urea, added to free nitrous acid (or a nitrite + dilute H_2SO_4), gives off nitrogen and carbon dioxide,

$$CO(NH_2)_2 + 2HNO_2 = CO_2 + 3H_2O + 2N_2$$

(complete on heating).

"*Nascent*" *hydrogen*, *e.g.* from zinc (coated with copper) and caustic potash, evolves ammonia from either nitrates or nitrites,

$$Zn + 2KHO = ZnO . K_2O + H_2$$

and

$$KNO_2 + 3H_2 = KHO + H_2O + NH_3.$$

Potassium permanganate + dil. H_2SO_4 is at once decolourized.

Meta-diamido-benzene $C_6H_4(NH_2)_2$ in presence of dilute H_2SO_4, a yellow colour, even in very dilute solutions

Aniline sulphate (one or two drops of solution) triturated with a concentrated solution of a nitrite gives the odour of phenol C_6H_5OH.

ACETATES.

(*E.g.* $CH_3 . COONa$.) Heated with concentrated H_2SO_4, acetic acid is given off (recognized by its odour)

$$CH_3 . COONa + H_2SO_4 = NaHSO_4 + CH_3 . COOH.$$

Heated with concentrated H_2SO_4 and *alcohol* (one or two drops) the characteristic odour of ethyl acetate is produced,

$$CH_3 . COOH + (C_2H_5)OH = CH_3 . COO(C_2H_5) + H_2O.$$

Heated to redness with arsenious anhydride, characteristic (poisonous) odour of cacodyl $As_2(CH_3)_4$ is produced.

Neutral solutions of acetates give with

Ferric chloride, a red colour, due to ferric acetate $(CH_3 . CO_2)_6Fe_2$. On boiling the solution, a brown ppt. is produced of basic ferric acetate $(CH_3 . CO_2)_6Fe_2 . nFe_2O_3$. The red colour is bleached by HCl but not by $HgCl_2$.

FORMATES.

(*E.g.* $H.COONa$.) Heated with concentrated H_2SO_4, carbon monoxide is evolved without blackening of the liquid,

$$H.COONa + H_2SO_4 = NaHSO_4 + H_2O + CO.$$

Neutral solutions of formates give with

Silver nitrate, on heating, a black ppt. of metallic silver.

Mercuric chloride, on heating, a white ppt. of mercurous chloride, or a grey ppt. of metallic mercury, according to quantity.

Ferric chloride gives a reaction similar to that produced with acetates.

PRELIMINARY EXAMINATION OF SOLID IN THE DRY WAY.

	Experiment	Observation		Inference
1.	Heat on charcoal before the blowpipe	Residue which is white and fusible (see also 5)		Alkali or alkaline earth
		" " " " infusible (see 2, 3 and 5)		
		" " yellow while hot, white when cold (see also 3)		Zn
		" " black or coloured, or metal beads (see 2 and 4)		
		Volatilizes (see also 2 and 6)		Hg, As, (NH_4), certain chlorides, organic acids, iodine, &c.
		Burns away		S, C, &c.
		Deflagrates		Nitrates, Chlorates, &c.
2.	Mix substance with Na_2CO_3* and again heat on charcoal in reducing flame	Red malleable metallic spangles or globules (see 4 and 5)		Cu
		Metal beads which are malleable, with yellow incrustation		Pb
		" " " without incrustation		Ag
		" " " with slight incrustation, yellow when hot		Sn
		" " brittle, with orange or yellow incrustation		Bi
		" " " with white incrustation		Sb
		Brown incrustation		Cd
		Odour of garlic (see also 6)		As
		" ammonia (see also 6)		NH_4
3.	Treat the white residue from 1. with cobalt nitrate and again heat	Blue mass		Al (possibly phosphates of Ca, &c.
		Bright green mass		Zn
		Dirty bluish green mass		Sn
		Pale pink mass		Mg
4.	Heat *small* quantity of substance in a borax bead, first in reducing, afterwards in oxidizing flame	**Reducing flame**	**Oxidizing flame**	
		Green	Green	Cr‡
		Blue	Blue	Co
		(Red†)	Green hot, blue cold	Cu
		Colourless	Violet	Mn‡
		Grey	Brownish violet	Ni
		(Bottle green)	Reddish yellow hot, pale yellow cold	Fe
5.	Heat substance moistened with HCl, on a clean platinum wire in reducing flame (or edge of Bunsen flame)	Flame is coloured Crimson		Sr
		" " Green		Ba, HBO_2, Cu
		" " Violet		K
		" " Intense yellow		Na
		" " Orange		Ca
		" " Blue		As, Sb, &c.
6.	Heat in dry bulb tube with dry Na_2CO_3 and powdered charcoal	Black mirror, which when heated in open tube gives white crystalline deposit		As
		Grey sublimate uniting into globules when rubbed		Hg
		Odour of ammonia		NH_4

* *In some cases, e.g. with tin compounds, it is better to use a mixture of Na_2CO_3 and KCy.*

† *Especially if a little tin be added.*

‡ *Confirm by fusing with excess of Na_2CO_3 and a little KNO_3. Mn bluish-green, Cr yellow.*

TO DISSOLVE A SUBSTANCE FOR ANALYSIS.

Boil the finely powdered substance with (1) water, (2) hydrochloric acid, (3) nitric acid, (trying both dilute and strong acids), (4) aqua regia; pouring off the previous solvent in each case. If a part only dissolves, the solution may be examined separately[*].

If all the above reagents fail to dissolve the substance[†], fuse it (or that portion which is insoluble) with about 5 times its weight of pure dry sodium carbonate[‡], extract the fused mass with boiling water[§].

Filter

Residue	Solution
Wash; dissolve in hot dilute HCl (or in presence of Ag and Pb, HNO_3) and test for metals (and silica) as usual[‖]. An insoluble residue is probably due to insufficient fusion.	Examine a portion for acids in the usual way, after acidifying with dilute HNO_3. Acidify the remainder with HCl and test as usual for silica and metals (Al, Cr, Mn, Zn, As, Sn).

Since cyanides, ferrocyanides, cobalticyanides, &c. often greatly interfere with the ordinary processes of analysis, it is best to destroy them, if present, before proceeding.

This may be done by evaporating to dryness with strong sulphuric acid, and heating, whereby the metals, including those in the acid cyanogen radicle, are obtained as sulphates.

In some cases silver nitrate in excess may be used, followed by excess of nitric acid. In this way the cyanides, &c. are obtained as silver salts, and the metals remain in solution as nitrates.

The solution for analysis is assumed to be neutral or slightly acid. If strongly acid it must be diluted largely, or nearly neutralized with Na_2CO_3. If *alkaline* it should be just acidified with dilute HNO_3, any precipitate produced being filtered off and examined separately.

* If the substance is a *metal* or *alloy*, heat it with strong nitric acid (adding water if necessary), evaporate to small bulk to remove excess of acid, add water and filter.

<table>
<tr><th colspan="2">Residue</th><th>Solution</th></tr>
<tr><td colspan="2">Contains antimony and tin [as hydrated SnO_2 and Sb_2O_5, or as arsenates; also possibly Bi as arsenate or phosphate; Au and Pt].
Digest with zinc and dilute HCl. Test evolved gas for AsH_3 by the $AgNO_3$ reaction. Residue on the zinc consists of metallic Sb and Sn (Bi, Au, Pt).
Boil for some time with strong HCl.</td><td rowspan="3">Examine for metals as usual, except for Sn, Sb (Au, Pt).</td></tr>
<tr><th>Residue Sb (Bi, Au, Pt).</th><th>Solution</th></tr>
<tr><td>Digest with yellow ammonium sulphide. Sb dissolves as sulphosalt.
Evaporate solution just to dryness.
Orange red residue ANTIMONY.</td><td>Contains Sn as stannous chloride.
Add mercuric chloride.
White ppt. (which usually turns grey on heating) TIN.</td></tr>
</table>

† The only common substances likely to remain insoluble in water and acids are:—SiO_2 and silicates. Sulphates of Ba, Sr, Pb (Ca). Fluoride of Ca, &c. Chrome iron ore. SnO_2. Sb_2O_4 and Sb_2O_5. AgCl (and some other Ag salts which are converted into AgCl). Ignited Sesquioxides of Fe, Al. Cr (in part). Ignited lead chromate. Some metaphosphates, arsenates, &c. Carbon. Sulphur. These are all more or less decomposed, or rendered soluble, by fusion with Na_2CO_3. Chrome iron ore is best decomposed by fusion with about 8 parts of Na_2CO_3 and 2 of powdered fused borax (or with a mixture of $2NaF+NaHSO_4$). Tin and antimony oxides may also be decomposed by fusion with KCy, whereby they are obtained as metals.

‡ A platinum crucible or foil may be used in absence of easily reducible metals (generally indicated in preliminary examination) such as Pb or Ag. If they are present, porcelain must be employed (in which case small quantities of Al, Si, &c. will be introduced), or else they must be first removed. $PbSO_4$ may be dissolved out with ammonium acetate and ammonia; AgCl with potassium cyanide.

§ The action of sodium carbonate here is, generally speaking, to give an insoluble carbonate or oxide of the metal, and a soluble sodium salt of the acid—*e.g.*

$$Na_2CO_3+BaSO_4=BaCO_3+Na_2SO_4. \qquad Na_2CO_3+CaF_2=CaCO_3+2NaF.$$

‖ In case of silicates, potassium and sodium must be looked for in a separate portion of the original substance. This may be done as follows—Heat the finely powdered substance with ammonium or calcium fluoride (pure) and strong H_2SO_4 (in a platinum vessel). In this way the silicon is removed as gaseous tetrafluoride.

$$4NH_4F+SiO_2+2H_2SO_4=2(NH_4)_2SO_4+2H_2O+SiF_4,$$

and the metals remain as sulphates. Extract with water and test solution for K and Na in the ordinary way (after removing previous groups, if present, as usual).

Or 1 part of the finely powdered substance may be strongly heated in a covered platinum vessel with about 8 parts of pure $CaCO_3$ and 1 part of NH_4Cl for about an hour. The mass is then extracted with water, and the calcium removed from the solution by precipitation with ammonium carbonate.

EXAMINATION FOR ONE METAL.

If solid, dissolve in (1) water, (2) HCl, (3) HNO_3 (try both dilute and strong acids), (4) aqua regia. Otherwise fuse with Na_2CO_3.
If solution is alkaline, just acidify with HNO_3 (dilute), filter off any ppt., and examine it separately. [See p. 42.]
If solution is strongly acid, dilute, or nearly neutralize it, before Group 2.
Test portions of the solution as follows:

1.	Add dilute *Hydrochloric Acid.*		Pour off liquid and shake ppt. with ammonia.		CONFIRMATORY TESTS in original solution or substance.
	White ppt. indicates*	SILVER	Dissolved		(1) K_2CrO_4 crimson ppt. (2) The HCl ppt. turns violet on exposure to light.
		LEAD	Unchanged		(1) Dilute H_2SO_4 white ppt. (2) K_2CrO_4 yellow ppt. soluble in KHO. (3) KI yellow ppt. soluble in boiling water.
		MERCURY Mercurous	Blackened		(1) Heat solid in bulb-tube with dry Na_2CO_3. Grey sublimate of metallic mercury. (2) KHO black ppt. insoluble in NH_3. (3) Same as for Mercuric (1).
2.	Add dilute HCl and pass *Hydrosulphuric Acid* gas. Heat.		Wash ppt. with water, and digest portions of it with hot *Ammonium sulphide*‡	*Ammonium carbonate*	
	Ppt. indicates†	ARSENIC (yellow)	Dissolved	Dissolved	(1) Try Marsh's test. Black stain on porcelain, soluble in NaClO. (2) Distinguish between As‴ and Asᵛ by adding $AgNO_3$ (in neutral solution) yellow = As‴, brick-red = Asᵛ.
		ANTIMONY (orange)	Dissolved	Undissolved	(1) Acidify dilute solution with HCl, pour it on to piece of zinc in contact with platinum foil; black stain on the platinum. (2) Marsh's test; black stain not dissolved by NaClO. [(3) Distinguish between Sb‴ and Sbᵛ by $AgNO_3$ test.]
		TIN (*-ous* dark brown) (*-ic* dirty yellow)	Dissolved	Undissolved	(1) Add small piece of zinc + dilute HCl. Allow to stand. Pour off liquid, and dissolve residue in strong hot HCl. Add $HgCl_2$. White ppt. (often turning grey on heating). (2) Distinguish between Sn″ and Snⁱᵛ by adding $HgCl_2$ to original solution. White ppt. Sn″. No ppt. Snⁱᵛ.
		MERCURY Mercuric (black)	Undissolved		(1) Acidulate with HCl and add metallic copper. [Nitric acid must be absent.] Bright deposit of metallic mercury on the copper. Wash, dry, and heat in dry test-tube. Grey sublimate of Hg. (2) KHO yellow ppt. (3) Dry test same as for Mercurous (1).
		LEAD (black)**	Ditto		See Group 1.
		COPPER (brownish-black)	Ditto (nearly)		(1) NH_3 pale blue ppt. redissolving at once in excess to deep blue solution. (2) K_4FeCy_6 chocolate ppt.
		BISMUTH (brownish-black)	Ditto		(1) NH_3 white ppt.; dissolve it in least possible HCl, and add much water; white milky ppt. (2) $SnCl_2$ + KHO black ppt. (3) K_2CrO_4 yellow ppt. insoluble in KHO.
		CADMIUM (yellow)	Ditto		(1) KCy in excess + H_2S yellow ppt. (2) NH_3 white ppt. easily soluble in excess.
	Add a few drops of nitric acid and boil. Then add NH_4Cl in excess, and *Ammonia*. Boil.		Test a *nitric* acid solution of original substance (or of the NH_3 ppt.) with *ammonium molybdate* in excess. Yellow ppt. on heating shews a PHOSPHATE. Test original solution with *Caustic Potash.*		
		ALUMINIUM§ (white)	White gelatinous ppt.; *soluble* in excess; repptd. by NH_4Cl		Heated on charcoal usually gives white infusible mass. Treated with $Co(NO_3)_2$ and again heated, blue mass.

Unless the contrary is stated, each reagent must

For explanation of the reactions which occur refer to General

Table, p. 47, and to special reactions of each metal.

3.	Ppt. indicates	CHROMIUM§ (bluish-green)	Green ppt.; soluble in excess; repptd. by boiling†	(1) Fuse with $Na_2CO_3+KNO_3$ on platinum foil. Yellow mass. Dissolve in water, add acetic acid and lead acetate. Yellow ppt. (2) Distinguish between Cr''' and Cr^{vi} by lead acetate test in original solution. [See Acids.]
		IRON§ (reddish-brown)	Reddish-brown ppt. *Ferric* } insoluble Dirty green ppt. *Ferrous* } in excess	(1) K_4FeCy_6 {pale blue ppt. Ferrous / dark blue ppt. Ferric . (2) KCyS {no colour. Ferrous / blood red colour. Ferric . (3) $K_6Fe_2Cy_{12}$. Dark blue ppt. Ferrous. No ppt. Ferric‖.
		PHOSPHATE of Ba, Sr, Ca, or Mg (white)	White ppt. insoluble in excess	Dissolve original substance (or NH_3 ppt.) in least possible quantity of HCl. Add large excess of sodium acetate and some acetic acid. [A ppt. here indicates Oxalate or Fluoride of Ca(BaSr), or phosphate of Fe(CrAl).] Add a few drops of Fe_2Cl_6; yellowish-white ppt. confirms Phosphate of Ca Ba Sr Mg. In this case add Fe_2Cl_6 until solution is red. Boil. Add NH_3. Filter. Test filtrate for Ca Ba Sr Mg by 5 and 6.
		OXALATE of Ca(BaSr) (white)	White ppt. insoluble in excess	Heat original substance (or NH_3 ppt.) to low redness. Add acetic acid. Effervescence indicates Oxalate. Filter if necessary. Test solution for Ca (Ba, Sr) by 5.
		FLUORIDE [or BORATE, &c.] of Ca(BaSr) (white)	White ppt. insoluble in excess	Test for Fluoride by heating with strong H_2SO_4. See Acids. Fuse original substance with Na_2CO_3. Extract fused mass with water. Filter. Test residue for Ca, &c., and solution for Fluoride, Borate, &c.
		SILICA (white)		Acidify with HCl. Evaporate to dryness. Digest residue with HCl. Residue insoluble in HCl = Silica.
4.	Add NH_4Cl, NH_3 and *Ammonium sulphide.*		Test original solution with *caustic potash.*	
	Ppt. indicates	NICKEL (black)	Pale green ppt. insoluble in excess	(1) Borax bead brownish-violet in oxidizing flame. (2) Add KCy until ppt. formed is just redissolved. Boil for some time in open dish. Add NaClO and allow to stand. Black ppt.
		COBALT (black)	Blue ppt. insoluble in excess	(1) Borax bead blue in both flames. (2) Add KCy, &c., as for Nickel. NaClO no ppt.
		MANGANESE (buff or pink)	White ppt. insoluble in excess. Rapidly turning brown when shaken with air	(1) Fuse with $Na_2CO_3(+KNO_3)$ on platinum foil. Bluish-green mass. (2) Distinguish between Mn'' and Mn''', Mn^{iv}, Mn^{vi}, &c., by boiling with HCl {Cl evolved—Mn''', Mn^{iv}, Mn^{vi}, &c. / no effect—Mn''.
		ZINC (white or greenish)	White ppt. *soluble* in excess	(1) Pass H_2S into the KHO solution. White ppt. (2) Heat on charcoal. Usually, yellow mass when hot, white when cold. Add $Co(NO_3)_2$ and again heat. Green mass.
5.	Add NH_4Cl in excess, NH_3 and *Ammonium carbonate.*		Test original solution with Calcium Sulphate	
	White ppt. indicates	BARIUM	Immediate white ppt.	(1) K_2CrO_4 yellow ppt. (2) H_2SiF_6 translucent crystalline ppt. (3) Flame test, green.
		STRONTIUM	White ppt. on standing or heating.	(1) Flame test, crimson. (2) H_2SiF_6, and K_2CrO_4 in dilute solution, no ppts.
		CALCIUM	No ppt.	(1) (In absence of Ba and Sr) Ammonium oxalate + NH_3 white ppt. Soluble in HCl; insoluble in acetic acid. (2) Flame test, orange red.
6.	Add NH_4Cl, NH_3 and *Sodium phosphate.* Stir. White ppt. MAGNESIUM. (Crystalline from dilute solutions, or on standing.)			
7.	Test portions of original solution for		AMMONIUM	(1) Boil with KHO. Odour of NH_3 produced. (2) $PtCl_4$ yellow ppt. (in lines on stirring). (See Potassium.)
			POTASSIUM	(1) (In absence of NH_4.) Acidify strong solution with HCl. Add $PtCl_4$. Stir. Yellow ppt. in lines. (2) Flame test, violet.
			SODIUM	(1) Flame test, intense yellow. Fixed residue on heating.

* A white ppt. which dissolves at once in cold water probably indicates a *Barium* salt. One which readily redissolves in slight excess of HCl probably *Antimony* [or *Bismuth*.

† { A white or nearly white ppt. which does not settle is probably Sulphur, indicating an *oxidizing agent.* If a ppt. of Sulphur is obtained, with much change of colour, filter off the ppt., boil filtrate to expel H_2S, and proceed with this, instead of original solution. An orange solution changing to green with pptn. of sulphur indicates Chromium as Cr^{vi}.

‡ Yellow. § As phosphate or otherwise. ‖ Ferrous salts almost always contain ferric.

** Lead is sometimes precipitated as *red* sulphochloride if much free HCl is present.

be added until *no further effect* is produced.

GENERAL TABLES FOR MIXTURES OF METALS.

(1)	(2)	(3)§	(4)	(5)§§
Add *Hydrochloric Acid* (dil.). Ppt. = { SILVER LEAD MERCURY (monad) Filter. Examine ppt. by Table I. and solution by column (2). A ppt. which redissolves at once in excess of HCl is probably due to Antimony or Bismuth. One which redissolves at once in cold water, is probably a Barium salt. Boric, Uric and Benzoic acids may be pptd. here if strong acid is used.	Pass *Hydrosulphuric Acid* gas to saturation, and heat*. Ppt. = † { MERCURY (dyad) LEAD BISMUTH COPPER CADMIUM } A ARSENIC ANTIMONY TIN } B Filter. Examine filtrate by column (3). Wash ppt. well and digest it for some time with hot yellow ammonium sulphide‡. Filter. RESIDUE (A) Examine by Table II. A. | SOLUTION (B) Examine by Table II. B.	Boil till H_2S is entirely expelled. Add nitric acid drop by drop as long as any change occurs. Then add NH_4Cl and *ammonia* till alkaline. Boil ‖. Ppt. = ¶ { IRON ALUMINIUM CHROMIUM } Hydroxides or Phosphates Barium Strontium Calcium Magnesium } Phosphates Calcium (Strontium Barium) } Oxalates (or Fluorides) Sometimes Manganese and Zinc in small quantities. Filter. Examine filtrate by column (4) and ppt. by Table III.	Add *Ammonium Sulphide* in slight excess and heat**. Ppt. = ‡‡ { ZINC MANGANESE †† NICKEL COBALT Filter. Examine filtrate by column (5) and ppt. by Table IV.	Add *Ammonium Carbonate* and heat gently. Ppt. = { BARIUM STRONTIUM CALCIUM Filter. Examine filtrate by Table VI. and ppt. by Table V.

---

* If much acid has been added, the solution should be diluted largely with water, or nearly neutralized, before passing H_2S. If a further pptn. occurs on heating, boil and pass H_2S again.

† A white or nearly white ppt. which does not settle is probably *sulphur*, shewing the presence of an oxidizing agent. If the solution turns bright green with pptn. of sulphur, chromic acid is probably present.

‡ Since CuS is somewhat soluble in yellow ammonium sulphide, it is advisable to use *sodium* sulphide if traces of Cu are being looked for.

§ Test a portion of this solution by evaporating to dryness and heating the residue. A residue which does not dissolve in HCl on heating is probably SILICA. If present, it must be removed from the remainder of the solution in the same way before proceeding. If the residue blackens considerably on heating, organic bodies such as sugar or tartaric acid are probably present. These should be destroyed by ignition, since they prevent the precipitation of this group, wholly or in part.

‖ If complete separation is required, the ppt. produced by ammonia must be redissolved in HCl and reprecipitated by NH_4Cl and NH_3, and this process repeated until the filtrate gives no ppt. with ammonium sulphide. In presence of chromium it is necessary to boil for a considerable time to ensure its precipitation with ammonia.

¶ Phosphates of zinc, manganese, nickel and cobalt may also be precipitated here, in part. If it is required to look for them, the ammonia ppt. must be washed and digested with ammonium sulphide, the filtrate tested for phosphate, and the ppt. washed and redissolved in HCl (with a drop of HNO_3 if necessary) and treated as in column (3). It is the safest plan always to treat the precipitate in this manner.

** It is advisable to test a few drops of the filtrate with ammonium sulphide before adding it to the main portion.

†† If a large excess of ammonia is present, manganese may be entirely held in solution, until the solution is boiled for some time.

‡‡ Possibly also some IRON, if much alkaline phosphate is present.

§§ If the filtrate from the ammonium sulphide ppt. is dark coloured (probably indicating nickel), the solution should be acidified with dilute HCl, boiled, filtered, and again rendered alkaline with NH_3, before proceeding.

EXPLANATION OF GENERAL TABLE.

Hydrochloric acid precipitates silver, lead (in part) and mercury (mercurous) as chlorides, $AgCl$, $PbCl_2$ and Hg_2Cl_2, thus separating them from all the other common metals, whose chlorides are soluble. (Bismuth and Antimony are often precipitated here as oxychlorides $BiOCl$ and $SbOCl$, but the ppt. at once dissolves in more HCl. Strong HCl precipitates barium salts, these being insoluble in strong acids; cold water at once dissolves the ppt.)

These chlorides having been filtered off, Hydrosulphuric acid precipitates mercury (mercuric), lead, bismuth, copper, cadmium, arsenic, antimony and tin, as sulphides, HgS, PbS, Bi_2S_3, CuS, CdS, As_2S_3, Sb_2S_3 or Sb_2S_5, SnS or SnS_2, these being insoluble in dilute HCl.

Arsen*ic* compounds are usually first reduced, in part at any rate, to the arseni*ous* state with separation of sulphur, *e.g.*

$$As_2O_5 + 2H_2S = As_2O_3 + 2H_2O + 2S.$$

This operation is very slow, requiring continued boiling, and repeated saturations with H_2S, hence arsenic in this state is often overlooked. [Some prefer to reduce the arsenic to the arsenious state by boiling with SO_2 before passing H_2S, but this process is liable to lead to complications, such as precipitation of Ba, Pb, &c. as sulphates.]

A precipitate of sulphur on passing H_2S indicates the presence of an "oxidizing" agent, such as free chlorine, hypochlorites, chromic arsenic or strong nitric acids, ferric salts, &c., *e.g.*

$$H_2S + Cl_2 = 2HCl + \underset{\text{pptd.}}{S}.$$

The ppt. produced by H_2S is well washed, in order to remove the salts which are still in solution, and free HCl, and digested with yellow ammonium sulphide*.

This converts the sulphides of arsenic, antimony, and tin, into soluble sulpho-salts, such as $(NH_4)_3AsS_3$, $(NH_4)_3SbS_3$ and $(NH_4)_2SnS_3$. For instance,

$$3(NH_4)_2S + As_2S_3 = 2(NH_4)_3AsS_3.$$

The remaining sulphides are left undissolved, and are separated by filtration.

The filtrate from the H_2S precipitate is tested with more H_2S to ascertain whether the previous group is entirely precipitated. If this is the case, the excess of H_2S is removed by boiling, and a few drops of nitric acid added, in order to convert any ferrous salt which may be present into the ferric state (since ferrous salts are only partially precipitated by ammonia). Thus

$$6FeCl_2 + 2HNO_3 + 6HCl = 3Fe_2Cl_6 + 4H_2O + 2NO.$$

The brown colour which is usually observed being due to a compound of NO with unaltered ferrous salt.

Ammonium chloride and ammonia are now added. (NH_4Cl has the property of forming double chlorides with the chlorides of magnesium, manganese, &c. which are not precipitated by NH_3, and are thus kept out of this group. It also renders the precipitation of aluminium hydroxide by ammonia more complete.)

---

* The yellow "ammonium sulphide" used in laboratories is a mixture consisting chiefly of polysulphides of ammonium (such as $(NH_4)_2S_2$) and ammonium thiosulphate $(NH_4)_2S_2O_3$.

EXPLANATION OF GENERAL TABLE (*continued*).

Ammonia precipitates iron, chromium and aluminium from most of their combinations, as hydroxides, such as $Fe_2(HO)_6$, $Cr_2(HO)_6$ and $Al_2(HO)_6$, *e.g.*

$$Al_2Cl_6 + 6NH_3 + 6H_2O = Al_2(OH)_6 + 6NH_4Cl,$$

and by neutralizing the acid in which they are dissolved, precipitates many salts which are insoluble in water, *i.e.* phosphates of Fe, Cr, Al, Ca, Ba, Sr, Mg; oxalates (fluorides, borates, silicates, &c. in part) of Ca, Ba, Sr.

(Ferric and aluminium phosphates are very imperfectly precipitated in presence of much alkaline phosphate, and may therefore occur in the next groups.)

In addition to the above, ammonia partially precipitates phosphates of Zn, Mn, Ni, Co. If these are present in any quantity, it is necessary to decompose them by digesting the precipitate with ammonium sulphide, which converts them into sulphides, *e.g.*

$$Zn_3(PO_4)_2 + 3(NH_4)_2S = 2(NH_4)_3PO_4 + 3ZnS.$$

The solution is tested for phosphoric acid with magnesia mixture, which, if found, indicates phosphates of Zn, Mn, Ni, Co, or Fe. The residue containing the sulphides of these metals is dissolved in acids, and treated exactly as before with NH_4Cl, NH_3, &c. The precipitate thus obtained is free from Zn, Mn, Ni, and Co. The filtrate from it may be examined separately for these metals. If present they probably existed as phosphates.

The separation of groups 3 and 4 by ammonium chloride and ammonia is very imperfect, manganese and zinc especially being carried down with the 3rd group. It is therefore necessary, in cases of accuracy, to repeatedly redissolve the ppt. in HCl, and reprecipitate it with NH_4Cl and NH_3.

A more complete separation of Fe''', Cr, Al, from Zn, Mn, Ni, Co, is effected by means of barium carbonate, which ppts. only Fe, Cr, Al. This process, although it takes considerable time, is advisable in cases where accuracy is required, especially if traces of zinc have to be searched for.

To the filtrate from the ammonia precipitate, ammonium sulphide is added. This throws down zinc, manganese, nickel and cobalt as sulphides, ZnS, MnS, NiS, CoS, these being insoluble in alkaline solution. Excess of ammonium sulphide dissolves nickel sulphide to a considerable extent, forming a black solution, and must therefore be avoided. In some cases (as when nickel sulphide has been thus dissolved) it is advisable to destroy the excess of ammonium sulphide with a dilute acid before proceeding,

$$(NH_4)_2S_2 + 2HCl = 2(NH_4)Cl + H_2S + \underset{\text{pptd.}}{S}.$$

The filtrate from the ammonium sulphide precipitate contains only Ba, Sr, Ca, Mg, and alkali metals. Ammonium carbonate is added, and the solution heated to decompose carbamate, bicarbonate, &c. This precipitates barium, strontium and calcium carbonates, $BaCO_3$, $SrCO_3$, $CaCO_3$. The liquid must not be boiled, since the reaction then tends to reverse, *e.g.*

$$CaCO_3 + 2NH_4Cl = (NH_4)_2CO_3 + CaCl_2.$$

The filtrate, containing only Mg and alkalies, is examined by Table VI.

TABLE I.

Ag, Pb, Hg'.

Wash ppt. with cold water, then boil it for some time with a considerable quantity of hot water.

Filter

<table>
<tr><td colspan="2">Residue
Digest with ammonia. Filter.</td><td rowspan="2">Solution
Add potassium chromate.
Yellow ppt. insoluble in acetic acid.
LEAD.</td></tr>
<tr><td>Residue (black)
MERCURY.
Confirm by drying ppt. at gentle heat, mixing with dry Na_2CO_3 and heating in a bulb-tube.
Grey ring united into globules by rubbing with a rod.</td><td>Solution
Acidify with HNO_3, white ppt. turning violet on exposure to sunlight.
SILVER.</td></tr>
</table>

The precipitate produced by hydrochloric acid contains silver, lead, and mercurous chlorides $AgCl$, $PbCl_2$, Hg_2Cl_2.

The separation of these is based upon the fact that

(1) $PbCl_2$ is soluble in boiling water, $AgCl$ and Hg_2Cl_2 insoluble.

(2) $AgCl$ is soluble in ammonia, Hg_2Cl_2 insoluble.

The $PbCl_2$ is first extracted with boiling water, and confirmatory tests for lead applied to the solution so obtained.

The $AgCl$ is extracted from the residue by ammonia, from which it is reprecipitated unchanged by nitric acid (the latter merely neutralizing the ammonia).

Hg_2Cl_2 is converted by ammonia into a black substance $(NHg'_2H_2)Cl$ (chloride of dimercurous-ammonium) which remains undissolved, and is confirmed by the dry test for mercury.

TABLE II. A.

Hg″, Pb, Bi, Cu, Cd.

Wash well. Heat with moderately dilute nitric acid as long as any action is observed.

FILTER

<table>
<tr>
<td rowspan="3">RESIDUE (black*)
MERCURY (mercuric)
Confirm by drying at a gentle heat, mixing with dry Na_2CO_3 and heating in a bulb-tube.
Grey ring, united into globules by rubbing with a rod.</td>
<td colspan="3">SOLUTION
Add dilute H_2SO_4 and an equal volume of alcohol†.
FILTER</td>
</tr>
<tr>
<td rowspan="2">White ppt.
LEAD.
Confirm by dissolving the ppt. in ammonium acetate and adding potassium chromate.
Yellow ppt. sol. in KHO.</td>
<td colspan="2">Boil off the alcohol. Add ammonia in excess.
FILTER</td>
</tr>
<tr>
<td>Ppt.
Dissolve in the least possible quantity of HCl.
Add much water.
White milky ppt.
BISMUTH.</td>
<td>Solution.
If blue contains COPPER.
Confirm by acidifying a portion with acetic acid and adding potassium ferrocyanide. Chocolate ppt.
If copper is present, add KCy to the remainder of blue solution until colourless. Pass H_2S.
Yellow ppt.
CADMIUM.</td>
</tr>
</table>

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[*] It is best to heat the ppt. with water and add strong HNO_3 drop by drop till the action commences. If the acid be too strong, lead will be chiefly converted into sulphate and remain in the residue with mercury. The latter may also be converted into a white substance.

[†] It is advisable to test a small portion for lead in the first instance, since, if absent, sulphuric acid and alcohol need not be added to the remainder.

EXPLANATION OF TABLE II. A.

The residue insoluble in ammonium sulphide contains mercury, lead, bismuth, copper, and cadmium, as sulphides.

Their separation is effected upon the following principles:

(1) HgS is insoluble in dilute nitric acid*. The remainder dissolve, forming nitrates with separation of sulphur, *e.g.*

$$3PbS + 8HNO_3 = 3Pb(NO_3)_2 + 4H_2O + 2NO + 3S.$$

(2) Dilute sulphuric acid precipitates lead as sulphate $PbSO_4$ (more completely in presence of alcohol), leaving the remaining metals in solution.

(3) Ammonia throws down bismuth as hydroxide, $Bi(OH)_3$, and gives a characteristic blue colour to the solution if copper is present (due to a compound of cuprammonium hydroxide $N_2H_6Cu''(OH)_2$). The copper may be further confirmed by the ferrocyanide test.

Bismuth hydroxide dissolves in hydrochloric acid, forming the chloride $BiCl_3$, which is converted by water into the insoluble oxychloride BiOCl—

$$BiCl_3 + H_2O = BiOCl + 2HCl.$$

An excess of hydrochloric acid must be avoided in dissolving the bismuth hydroxide, since it prevents the formation of the oxychloride.

If copper is present potassium cyanide is added, which forms with it a colourless double cyanide $Cu_2Cy_2 . 2KCy$. Hydrosulphuric acid does not precipitate copper from this compound, whilst it throws down cadmium as yellow sulphide CdS.

Copper may also be separated from cadmium by precipitating the two together as sulphides, and boiling the precipitate with dilute H_2SO_4. CdS dissolves, CuS remains insoluble. Or, in absence of ammonium salts, by adding a strongly alkaline solution of sodium tartrate and boiling; cadmium is precipitated as oxide, copper remaining in solution.

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* Some prefer to use strong nitric acid, in which case the lead is converted into sulphate, and remains in the residue with HgS, the latter being often converted into a white substance $Hg(NO_3)_2 . 2HgS$. They may be then separated by ammonium acetate, which dissolves only lead sulphate.

TABLE II. B. METHOD 1.

Acidify with dil. HCl. Filter. Neglect filtrate. (If ppt. is nearly white and does not settle, it is probably only sulphur, and may be neglected.)

Wash ppt. and digest it for some time with warm ammonium carbonate*.

FILTER

<table>
<tr><th>SOLUTION</th><th colspan="2">RESIDUE</th></tr>
<tr><td rowspan="3">Acidify with HCl.
Yellow ppt.—probably
ARSENIC†.
Confirm by dissolving ppt. in HCl with a drop of HNO_3 and trying Marsh's test‡.
[Some Tin may also be present in this ppt. To confirm it, roast ppt. in open vessel, and fuse residue with KCy. Metallic Sn remains, which when dissolved in HCl gives white ppt. with $HgCl_2$.]</td><td colspan="2">Wash. Heat with strong HCl. Filter if necessary. (Residue is only S and traces of As_2S_3.) Heat gently to remove H_2S. Divide solution into two parts.</td></tr>
<tr><td>1.</td><td>2.</td></tr>
<tr><td>Test for Sn.
Add a *small* piece of zinc. Allow to stand. Pour off clear solution. Dissolve residue in strong hot HCl. Add $HgCl_2$. White ppt. often turning grey.
TIN†.</td><td>Test for Sb.
Pour the solution on to a piece of zinc on platinum foil. Black stain on the platinum.
ANTIMONY†.
Confirm by dissolving stain off in ammonium sulphide, and gently evaporating the solution.
Orange residue if Sb is present.</td></tr>
</table>

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* Preferably *sequi*-carbonate, saturated in the cold. Instead of ammonium carbonate, strong boiling HCl may be used, in which case Sn and Sb dissolve as chlorides, while As_2S_3 remains insoluble.

† Ascertain, if possible, whether *-ous* or *-ic* (or both) in original solution.

‡ Or by heating in a bulb-tube with dry Na_2CO_3, and charcoal or KCy.

EXPLANATION OF TABLE II. B. METHOD 1.

The solution after digestion of the mixed sulphides with yellow ammonium sulphide contains arsenic, antimony, and tin, as sulpho-salts.

These are decomposed by dilute HCl, the insoluble sulphides being reprecipitated, *e.g.*

$$\underset{\substack{\text{soluble ammonium}\\ \text{sulpharsenite}}}{3(NH_4)_2S\,.\,As_2S_3} + 6HCl = \underset{\substack{\text{insoluble}\\ \text{arsenious sulphide}}}{As_2S_3} + 6NH_4Cl + 3H_2S.$$

Tin will always be reprecipitated as yellow stann*ic* sulphide, since stannous sulphide is converted into stannic by the excess of sulphur in the yellow ammonium sulphide.

[A *white* ppt. is merely sulphur from the yellow ammonium sulphide,

$$(NH_4)_2S_2 + 2HCl = 2NH_4Cl + H_2S + S.]$$

The ppt. (As_2S_3 (or As_2S_5), Sb_2S_3 (or Sb_2S_5), SnS_2 and free S) is washed till free from HCl, and digested with warm ammonium carbonate.

As_2S_3 dissolves; the reaction may be approximately represented thus—

$$4As_2S_3 + 4(NH_4)_2CO_3 = \underset{\substack{\text{soluble}\\ \text{arsenite}}}{(NH_4)_2O\,.\,As_2O_3} + \underset{\substack{\text{soluble}\\ \text{sulpharsenite}}}{3\{(NH_4)_2S\,.\,As_2S_3\}} + 4CO_2.$$

Sb_2S_3 (and Sb_2S_5) are practically insoluble, and SnS_2 very slightly soluble.

The solution of arsenite and sulpharsenite is acidified with HCl, which precipitates the arsenic as As_2S_3.

$$(NH_4)_2O\,.\,As_2O_3 + 3\{(NH_4)_2S\,.\,As_2S_3\} + 8HCl = 4As_2S_3 + 8NH_4Cl + 4H_2O.$$

Arsenic is confirmed in this precipitate, by dissolving in acids, and trying Marsh's test; or else in the dry way.

The residue insoluble in ammonium carbonate, containing the antimony and tin, is dissolved in strong hot HCl. SnS_2 dissolves, forming $SnCl_4$; Sb_2S_3 and Sb_2S_5 both form $SbCl_3$ (the latter with separation of S); H_2S being evolved.

Sulphur and traces of As_2S_3 remain undissolved.

The HCl solution is divided into two parts.

One is examined for tin, by adding zinc. This precipitates tin as metal,

$$SnCl_4 + 2Zn = Sn + 2ZnCl_2.$$

The deposited tin is dissolved in HCl, which converts it into stann*ous* chloride $SnCl_2$, and mercuric chloride added. This is reduced first to white mercurous chloride, and afterwards, if sufficient stannous salt be present, to grey metallic mercury.

$$2HgCl_2 + SnCl_2 = Hg_2Cl_2 + SnCl_4$$

and

$$Hg_2Cl_2 + SnCl_2 = 2Hg + SnCl_4.$$

The other part is examined for antimony by causing the solution to act upon zinc in contact with platinum foil. Antimony is deposited as metal on the platinum. On digesting with yellow ammonium sulphide, antimony dissolves as a sulpho-salt, and on evaporation of the solution, orange antimony sulphide remains.

TABLE II. B. METHOD 2.

Acidify with HCl (dil.). Filter. Neglect filtrate. (If ppt. is nearly white, and does not settle, it is probably only sulphur, and may be neglected.)

Wash ppt. and dissolve it in strong HCl (with a drop of HNO_3, or a crystal of $KClO_3$, if necessary). Heat gently to expel chlorine, &c. Reserve a portion. Introduce remainder into a vessel in which hydrogen is being generated by the action of Zn and dil. H_2SO_4.

Pass the evolved gases through a wash-bottle containing solution of lead acetate, and then into a strong solution of silver nitrate. A black ppt. indicates As or Sb.

FILTER

SOLUTION	PPT.	
Add a few drops of $AgNO_3$ and then dilute ammonia, drop by drop. Yellow ppt. where the liquids meet, ARSENIC.	Wash well. Digest with hot tartaric acid on the filter.	
	SOLUTION	RESIDUE neglect.
	Add HCl dil. and pass H_2S. Orange ppt. ANTIMONY.	

Examine the other portion of the solution for tin, by adding a small piece of zinc, and allowing to stand. Pour off clear solution. Boil residue with strong HCl. Add $HgCl_2$. White ppt. often turning grey, TIN.

EXPLANATION OF TABLE II. B. METHOD 2.

This method is based upon the fact that compounds of arsenic and antimony when brought in contact with zinc and dilute acid, (HCl or H_2SO_4) evolve gaseous arsenic and antimony trihydrides, AsH_3 and SbH_3. (A portion of the As and Sb being usually precipitated on the Zn.)

The gases are purified from any H_2S which may be present by passing through a solution of lead acetate, and then led into a solution of silver nitrate. This decomposes them as follows:—

$$AsH_3 + 6AgNO_3 + 3H_2O = \underset{\text{Black ppt.}}{6Ag} + \underbrace{H_3AsO_3 + 6HNO_3}_{\text{in solution}}$$

$$SbH_3 + 3AgNO_3 = \underset{\text{Black ppt.}}{SbAg_3} + 3HNO_3.$$

The solution therefore contains arsenious acid, nitric acid, and excess of silver nitrate. On carefully neutralizing with ammonia, yellow silver arsenite Ag_3AsO_3 is precipitated. (This ppt. being soluble either in HNO_3 or NH_3, exact neutralization is necessary.)

The residue on the filter consists of metallic silver, and silver stibide Ag_3Sb. Tartaric acid dissolves Sb leaving Ag, and on passing H_2S into the solution, Sb_2S_3 is obtained as an orange precipitate. The Tin is found as in Method 1.

TABLE III.

Ppt. may contain Al, Fe, Cr, hydroxides or phosphates. Ca, Ba, Sr, Mg, phosphates. Ca(BaSr) oxalates (fluorides, borates)*. Sometimes Mn and Zn†.

Wash thoroughly.

1 Dissolve a small portion in HNO_3, add ammonium molybdate in excess, and heat. Yellow ppt. indicates a PHOSPHATE‡. [If phosphates are absent, those parts of this table in small print may be omitted.]

2 Fuse a portion with Na_2CO_3 (and a little KNO_3) on platinum foil. Bluish-green mass indicates MANGANESE. Yellow mass CHROMIUM. Confirm Cr by dissolving mass in water, acidulating with acetic acid, and adding lead acetate. Yellow ppt. shews Cr.

3 Dissolve remainder in the least possible quantity of HCl§. Test a small portion of the solution by diluting and adding K_4FeCy_6. Blue ppt. indicates IRON. To the remainder add KHO in large excess and boil. [If Cr is present, boil for a considerable time‖.]

FILTER

<table>
<tr><td colspan="2">SOLUTION</td><td colspan="3">PPT.</td></tr>
<tr><td colspan="2">Add NH_4Cl in excess and boil.
FILTER</td><td colspan="3">Wash. Dissolve in the least possible quantity of HCl. Add a strong solution of sodium acetate in excess, and some acetic acid.
FILTER</td></tr>
<tr><td>Solution</td><td>Ppt.</td><td>Ppt.</td><td colspan="2">Solution</td></tr>
<tr><td rowspan="3">*Test for phosphate by adding magnesia mixture. If found, was probably present as Ferric phosphate.*</td><td rowspan="3">indicates ALUMINIUM
hydroxide or phosphate. Wash and dissolve it in HNO_3 *and test for phosphoric acid as above with* $(NH_4)_2MoO_4$.</td><td rowspan="3">Oxalates (fluorides) of Ca(BaSr).
Possibly also phosphates of Fe *and* Cr, *which may be confirmed as above* 1, 2, 3.
Wash, dry, heat to low redness. Add acetic acid. Effervescence indicates an OXALATE‖. Test acetic solution for Ca, &c. as usual. Ppt.¶ may also be tested for FLUORIDE by heating with H_2SO_4.</td><td colspan="2">*Add* Fe_2Cl_6 *drop by drop. Whitish ppt. indicates PHOSPHATE of* Ca, Ba, Sr *or* Mg.
If a ppt. is produced, continue to add Fe_2Cl_6 *until the solution is red.*
Boil, filter while hot.</td></tr>
<tr><td>*Ppt.*</td><td>*Solution*</td></tr>
<tr><td>*neglect.*</td><td>*Add* NH_4Cl *and* NH_3. *Filter if necessary. Neglect ppt. Examine solution for* CaBaSrMg *as usual by Table V.*</td></tr>
</table>

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* Fluorides and Borates are only imperfectly precipitated in this group. The metals (Ca, Ba, Sr) will therefore be found in Group V. and the acids as usual.

† See note (‖) General Table.

‡ Arsenate would give a similar reaction, if not previously separated by H_2S.

§ If much chromium is present it is advisable, instead of dissolving the ppt. in HCl and adding KHO, to fuse it with Na_2CO_3 and KNO_3, extract with water, and filter. The Cr will remain in solution together with the Al, and will not interfere with the tests for the latter.

‖ Calcium oxalate may be decomposed to some extent by potash under these conditions, hence, if its presence be suspected, it is advisable to test for it in a portion of the original NH_3 ppt. instead of the ppt. produced by potash.

¶ In most cases the original substance may be used instead.

EXPLANATION OF TABLE III.

Since the presence of phosphates renders the analysis of this group more complex, it is advisable to test a portion of the precipitate at once for phosphate. This is done by dissolving in nitric acid (since hydrochloric acid impairs the delicacy of the reaction), adding ammonium molybdate, and heating, which gives a yellow precipitate of "phospho-molybdate of ammonium" (composition doubtful). It is necessary to employ a large excess of the molybdate, since excess of phosphate prevents the formation of the precipitate.

Fusion of the precipitate with sodium carbonate, in presence of air or some oxidizing agent such as KNO_3, converts lower oxides of chromium and manganese into a chromate and manganate respectively, *e.g.*

$$Cr_2O_3 + 2Na_2CO_3 + 3O = \underset{\text{yellow mass}}{2Na_2CrO_4} + 2CO_2$$

and

$$MnO + Na_2CO_3 + O_2 = \underset{\text{blue-green mass}}{Na_2MnO_4} + CO_2.$$

The fused mass is extracted with water. (If much manganate is present, a drop of alcohol may be added, which will precipitate the manganese as Mn_2O_3 (hydrated), leaving the chromate in solution.) Acetic acid is added to remove the excess of alkali, and lead acetate to precipitate the chromate as yellow $PbCrO_4$.

The remainder of the precipitate is dissolved in hydrochloric acid. The phosphates, oxalates, &c. dissolve potentially as such, whilst the hydroxides are converted into chlorides. A portion of the solution is tested for iron by potassium ferrocyanide, (which gives "Prussian blue" $Fe_4(FeCy_6)_3$* if iron is present,) and caustic potash added in excess to the remainder This reprecipitates the whole group substantially in the same condition as before, but redissolves aluminium hydroxide and phosphate, the former as potassium aluminate $Al_2(OK)_6$. Chromium hydroxide and phosphate also redissolve in excess of cold KHO, but are reprecipitated by continued boiling. If much chromium has been indicated however, it is best to convert it entirely into the hexad (or chromic acid) form before proceeding, by fusion with Na_2CO_3, &c. as above. In this condition it will remain in solution and will not interfere with the other reactions.

The filtered solution, after boiling with potash, is boiled with ammonium chloride. This decomposes the KHO as follows:

$$KHO + NH_4Cl = KCl + H_2O + NH_3.$$

Aluminium hydroxide and phosphate are consequently reprecipitated.

Boiling with KHO usually decomposes ferric phosphate, in part at any rate. Thus

$$2FePO_4 + 6KHO = Fe_2(OH)_6 + 2K_3PO_4.$$

If therefore, after the separation of aluminium, much phosphate is found in solution, ferric phosphate was probably present.

The precipitate produced by caustic potash is dissolved again in hydrochloric acid and excess of sodium acetate added. This has the effect of replacing free hydrochloric acid by free acetic acid:—

$$HCl + CH_3COONa = NaCl + CH_3COOH.$$

---

* See footnote, p. 11.

EXPLANATION OF TABLE III. (*continued*).

Calcium oxalate and fluoride, ferric and chromic phosphates, are soluble in hydrochloric acid, but insoluble, or nearly so, in acetic acid, and are therefore precipitated. [Oxalates are confirmed by heating the precipitate, which converts them into carbonates. Fluorides by heating with H_2SO_4, whereby HF is evolved; and ferric and chromic phosphates by testing as above for Fe, Cr, and PO_4.]

Phosphates of Ca, Ba, Sr and Mg, however, are soluble both in hydrochloric acid and in acetic acid, and therefore remain in solution. The addition of ferric chloride decomposes them as follows:

$$Ca_3(PO_4)_2 + Fe_2Cl_6 = 2FePO_4 + 3CaCl_2.$$

If, therefore, a reddish white ppt. is obtained on the addition of the first few drops of Fe_2Cl_6, it proves that phosphate of Ca, Ba, Sr, or Mg, was present. In this case Fe_2Cl_6 must be added until the solution is red, shewing that the decomposition of Ca &c. phosphates is complete. The phosphoric acid is thus entirely got rid of, leaving the Ca, Ba, Sr, Mg, as chlorides, in which form they can be identified as usual.

Oxalates and Phosphates of Ca, Sr, Ba, may also be analysed by dissolving in the least possible quantity of HCl, adding dilute H_2SO_4 and about $\frac{1}{3}$ total volume of alcohol. After standing some hours the metals are obtained as sulphates and the solution contains oxalic and phosphoric acids.

TABLE IV.

NiS, CoS, ZnS, MnS.

Wash; digest with cold dilute hydrochloric acid.

FILTER

RESIDUE (if not black, neglect)

Examine a portion with the borax bead.

Blue bead in both flames indicates Co.

Brownish-violet in oxidizing flame } Ni.
Grey in reducing flame (after a time) } Ni.

Dissolve remainder in the least possible quantity of HCl with a drop of HNO_3. (Filter off S if necessary.)

Nearly neutralize with KHO. Add solution of (pure) KCy till the ppt. which is first formed is just redissolved. Boil for several minutes in an open dish. Filter off any slight ppt., and add a clear solution of HgO in $HgCy_2$*. Boil.

FILTER

PPT. (pale green)	SOLUTION
NICKEL Wash thoroughly and confirm by borax bead.	Evaporate to dryness and test residue for COBALT by borax bead.

SOLUTION†

Boil to expel H_2S. Cool, and add KHO *in excess* without boiling.

FILTER

PPT.	SOLUTION
Fuse with Na_2CO_3 (and a little KNO_3) on platinum foil. Blue-green mass. MANGANESE.	Pass H_2S. White (or nearly white) ppt. ZINC. (May be further confirmed by heating ppt. on charcoal with cobalt nitrate. Green mass.)

---

* Prepared by boiling freshly precipitated HgO in strong solution of $HgCy_2$ and filtering. (NaClO, or bromine + KHO, may be used instead, but solution must not then be boiled.

† Some IRON may also be present in this solution if much alkaline phosphate was originally present. Test a few drops therefore with potassium ferrocyanide.

EXPLANATION OF TABLE IV.

Zinc and manganese sulphides dissolve readily in cold dilute hydrochloric acid (forming chlorides $ZnCl_2$ and $MnCl_2$, with evolution of H_2S); whilst nickel and cobalt sulphides are only dissolved in traces which do not interfere with the detection of zinc and manganese.

The residue consisting of NiS and CoS is examined with the borax bead. If only nickel is indicated, it is not usually necessary to examine further, but if cobalt is found, the process of separation of nickel and cobalt must be gone through, since the blue given to the borax bead by cobalt may completely mask the nickel colour. It is safer however always to go through the entire process in either case, especially if small quantities have to be looked for.

In order to separate nickel and cobalt, the sulphides are dissolved in aqua regia, whereby they are converted into chlorides $NiCl_2$ and $CoCl_2$. The solution is nearly neutralized to remove the excess of free acid, and potassium cyanide added, which precipitates the single cyanides $NiCy_2$ and $CoCy_2$. These dissolve in excess of potassium cyanide, forming soluble double cyanides $NiCy_2 2KCy$, and $CoCy_2 2KCy$.

On boiling the solution in presence of air, the latter reacts with the excess of KCy which is present, and oxygen, thus

$$2(CoCy_2 2KCy) + 4KCy + H_2O + O = K_6Co_2Cy_{12} + 2KHO,$$

[or if free acid be present,

$$2(CoCy_2 2KCy) + 2KCy + 2HCy + O = K_6Co_2Cy_{12} + H_2O],$$

$NiCy_2 2KCy$ remaining unaltered.

$K_6Co_2Cy_{12}$ is tolerably stable, and is not acted upon by mercuric oxide (or oxycyanide*), or by sodium hypochlorite at a gentle heat; whereas $NiCy_2 . 2KCy$ is readily decomposed by these reagents as follows:

$$NiCy_2 + HgOHgCy_2 + H_2O = \underset{\text{Pale green ppt.}}{Ni(OH)_2} + 2HgCy_2,$$

$$2NiCy_2 + NaClO + 5H_2O = \underset{\text{Black ppt.}}{Ni_2(OH)_6} + NaCl + 4HCy.$$

The solution is examined for cobalt by evaporating to dryness and testing with the borax bead [or by neutralizing with dilute HNO_3 and adding mercurous nitrate, whereby white $Hg_6Co_2Cy_{12}$ is precipitated. This on ignition leaves black Co_3O_4].

---

* It is preferable to use mercuric oxide dissolved in mercuric cyanide (*i.e.* mercuric oxycyanide) since it can be added as a *solution*. If mercuric oxide alone be used, the excess of HgO masks the precipitate of $Ni(OH)_2 . NiCy_2$ which is then produced.

EXPLANATION OF TABLE IV. (*continued*).

To the solution containing $ZnCl_2$ and $MnCl_2$, caustic potash is added. This throws down $Zn(OH)_2$ and $Mn(OH)_2$ as white precipitates[*]. $Zn(OH)_2$ is soluble[†] in excess of cold KHO, forming ZnK_2O_2 (Potassium zincate), $Mn(OH)_2$ remaining insoluble. [If the solution be heated, $Zn(OH)_2$ will often not redissolve in KHO, especially if dilute.]

H_2S passed into the solution precipitates white zinc sulphide,

$$K_2ZnO_2 + 3H_2S = ZnS + 2KHS + 2H_2O.$$

(This precipitate is often discoloured owing to traces of impurity.)

The precipitate produced by KHO which does not redissolve in excess is examined for manganese by fusing with sodium carbonate in presence of air (or some oxidizing agent as KNO_3),

$$Mn(OH)_2 + Na_2CO_3 + O_2 = \underset{\text{Blue green mass}}{Na_2MnO_4} + H_2O + CO_2.$$

Manganese may also be separated from nickel, cobalt, and zinc, by passing hydrosulphuric acid into an *acetic acid* solution (containing no other free acid); NiS, CoS, and ZnS, being insoluble in acetic acid, are precipitated, whilst Mn remains in solution.

Nickel may be separated from cobalt by several other methods; for instance:—

By means of *potassium nitrite* and acetic acid in a neutral solution, which precipitates all the cobalt, after a time, as yellow $K_6Co_2(NO_2)_{12} . 3H_2O$, nickel remaining in solution.

Or by precipitating the metals as *ferricyanides* (in presence of ammonium chloride), and digesting the precipitate with ammonia. Nickel ferricyanide dissolves, cobalt ferricyanide remains.

---

[*] The white $Mn(OH)_2$ rapidly turns brown owing to absorption of oxygen and formation of $Mn_2O_3 . H_2O$.

[†] Incompletely in presence of Mn, Ni, Co.

TABLE V.

$BaCO_3$, $SrCO_3$, $CaCO_3$.

Dissolve the main portion of the ppt. in the least possible quantity of *dilute* HCl*.

Test a part of the solution for Ba and Sr by adding $CaSO_4$ and heating.

$\begin{cases}\text{Immediate ppt.} = \text{Ba,} \\ \text{ppt. after a time} = \text{Sr.}\end{cases}$

If no ppt. **even on standing, test remainder of solution at once for Ca as below†.**

If Ba or Sr are present, **evaporate remainder of solution to small bulk, add H_2SiF_6 in excess, and an equal volume of alcohol.**

FILTER

<table>
<tr><th>Ppt.</th><th colspan="2">Solution</th></tr>
<tr><td rowspan="2">BARIUM.
Confirm by dissolving another portion of the $(NH_4)_2CO_3$ ppt. in <i>acetic</i> acid, and adding potassium chromate.
Yellow ppt. = Ba.
Dissolve ppt. in HCl and try flame-test.</td><td colspan="2">To a <i>portion</i> add $CaSO_4$, heat, and allow to stand. White ppt. after a time STRONTIUM.
If Strontium is present, add dilute H_2SO_4 to <i>remainder</i> of solution, boil, and allow to stand some time.
FILTER</td></tr>
<tr><td>Ppt.
Neglect.</td><td>Solution†
Add NH_3 till alkaline (filter if necessary), and ammonium oxalate. White ppt.
CALCIUM.
Dissolve ppt. in HCl and try flame-test.</td></tr>
</table>

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* In most cases information can be obtained by trying the flame-test with a portion of this solution. Ba gives a green flame, Sr crimson, Ca orange-red.

EXPLANATION OF TABLE V.

On dissolving the precipitate of barium, strontium, and calcium carbonates in hydrochloric acid, the chlorides, $BaCl_2$, $SrCl_2$, and $CaCl_2$ are obtained, CO_2 being evolved. *Dilute* HCl must be used, since barium salts are insoluble in strong mineral acids.

Calcium sulphate is sufficiently soluble in water to be used as a test for barium and strontium, whose sulphates are practically insoluble. Sulphuric acid, or other sulphates, would precipitate calcium as well, from a strong solution.

Barium would answer the special tests about to be applied for strontium and for calcium, and, if present, must therefore be removed before these can be looked for. For the same reason, strontium must be removed before testing for calcium.

The barium is separated by means of hydrofluosilicic acid, which gives a translucent precipitate of $BaSiF_6$ (more complete in presence of alcohol), strontium and calcium remaining in solution. A separate portion of the original precipitate is dissolved in acetic acid, and potassium chromate added, in order to confirm barium, which gives yellow $BaCrO_4$, insoluble in acetic acid.

A portion of the filtrate from the H_2SiF_6 precipitate is tested for strontium with calcium sulphate. If present, dilute sulphuric acid is added to the remainder. This precipitates *all* the strontium and may precipitate *part* of the calcium, as sulphates. Enough calcium is always left in solution to be detected by the ammonium oxalate reaction, which gives, in neutral or ammoniacal solution, a white precipitate of calcium oxalate $(CO_2)_2Ca$.

Barium may also be separated from strontium and calcium by means of *potassium chromate*, which, in an *acetic acid* or *dilute neutral* solution, precipitates only $BaCrO_4$.

Also by converting the metals into *chlorides*, evaporating to dryness, and digesting with absolute *alcohol*. $SrCl_2$ and $CaCl_2$ dissolve, $BaCl_2$ remains insoluble.

Strontium may be separated from calcium by converting the metals into sulphates and digesting with a hot strong solution of *ammonium sulphate*. $CaSO_4$ dissolves, $SrSO_4$ remains insoluble.

Also by converting the metals into *nitrates*, evaporating to dryness, and digesting with absolute *alcohol*. $Ca(NO_3)_2$ dissolves, $Sr(NO_3)_2$ remains insoluble.

TABLE VI.

Solution may contain Mg, K, Na, NH_4.

Evaporate to dryness. Heat residue to low redness until fumes cease to be evolved.

1. Dissolve a portion of residue in water (and a drop of dil. HCl if necessary). Test the solution with NH_4Cl, NH_3 and sodium phosphate. Warm, shake, and allow to stand. White ppt. (crystalline from dilute solutions or on standing) = MAGNESIUM*.

2. Dissolve the remainder in the least possible quantity of water†. Filter if necessary. Test a portion of the solution by stirring on a watch glass with $PtCl_4$‡. Yellow ppt. in lines = POTASSIUM.

   Examine another portion by the flame-test on a *clean* platinum wire. Strong and persistent yellow colouration = SODIUM. Violet colouration POTASSIUM.

Test a portion of the *original* substance or solution for ammonium by boiling with potash. Fumes of ammonia (recognised by odour and action on red litmus paper) AMMONIUM.

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* A slight flocculent ppt. which does not become crystalline on standing may be due to Ba, Ca, Al, &c. In doubtful cases, therefore, add a mixture of ammonium sulphate, ammonium oxalate and ammonia, boil and filter before testing for Mg.

† If much Mg is indicated it should be removed before testing for K and Na. This may be done by adding $Ba(OH)_2$, filtering, removing the excess of Ba salt with ammonium carbonate, evaporating, and igniting to remove ammonium salts.

‡ If iodides are present, $PtCl_4$ will produce a strong red colour.

EXPLANATION OF TABLE VI.

Before testing for the remaining metals, it is necessary to remove all ammonium salts from the solution; [since $PtCl_4$ gives the same reaction with them as with potassium salts; and since the precipitation of magnesium by sodium phosphate is rendered less sensitive, and that by barium hydroxide prevented altogether, by their presence.] This is done by evaporating to dryness and igniting the residue, whereby ammonium salts being volatile, are expelled. Too high a temperature must not be used, since sodium and potassium chlorides might then be volatilized also.

A portion of the residue is dissolved in water (and a drop of dilute HCl if necessary, since $MgCl_2$ leaves a sparingly soluble oxychloride on evaporation of its solution) and tested for magnesium with NH_4Cl, NH_3 and Na_2HPO_4,

$$Na_2HPO_4 + NH_3 + MgCl_2 = (NH_4)MgPO_4 + 2NaCl.$$

[The NH_4Cl prevents the precipitation of $Mg(OH)_2$ by NH_3, owing to the formation of a soluble double chloride ($MgCl_2 . 2NH_4Cl$).]

The remainder of the residue is dissolved in water (filtered if necessary from $MgOMgCl_2$) and examined for potassium and sodium; the former by $PtCl_4$, which gives a yellow precipitate of K_2PtCl_6 (more complete in the presence of alcohol), and the latter by the flame-test, sodium compounds giving a strong yellow flame.

Acid potassium metantimoniate $K_2O . H_2O . Sb_2O_5 . 6Aq$ is sometimes employed as a test for sodium salts, with which it produces a white precipitate of $Na_2O . H_2O . Sb_2O_5 . 6Aq$. The solution to be tested must be neutral, and the reagent freshly prepared.

The separation of magnesium from alkali metals may be effected in several ways. In the process here recommended it is precipitated as $Mg(OH)_2$ by means of barium hydroxide; the barium being then removed as $BaCO_3$, by adding ammonium carbonate. Mercuric oxide may be used instead of barium hydroxide, and the mercury afterwards got rid of by heating to low redness.

Another method is to precipitate the magnesium by ammonium phosphate, and afterwards remove the excess of phosphate by ferric chloride.

Or the solution (containing the metals as chlorides) may be evaporated to dryness with ammonium oxalate and heated, whereby they are converted into carbonates. On digesting the residue with water, $MgCO_3$ remains insoluble.

PRELIMINARY EXAMINATION FOR ACID RADICLES.

1. Treat the solid substance (or strong solution) with *dilute Hydrochloric acid* in the cold. Afterwards heat.		CONFIRMATORY TESTS.
Effervescence *in the cold* with evolution CO_2 (detected by pouring the evolved gas into lime-water and shaking)	CARBONATE or Cyanate*	Further confirmation not usually required. If soluble, $BaCl_2$ or $CaCl_2$ gives white ppt. soluble in acids with effervescence. (N.B. Cyanides usually contain carbonates.)
„ „ „ with evolution of Cl, (detected by odour, colour, and action on KI paper) . . .	HYPOCHLORITE †	(1) $MnCl_2$, brown ppt. (2) Solution bleaches litmus, writing-ink, &c. (N.B. Hypochlorites always contain chlorides.)
Evolution of H_2S (detected by odour and action on lead acetate paper)	SULPHIDE‡	If soluble in water, (1) Sodium nitroprusside, violet colouration. (2) Silver coin stained black.
„ „ SO_2 (detected by odour, and by action on $K_2Cr_2O_7$§)	SULPHITE	(1) Zn + dil. HCl. H_2S evolved. Detected as above. (2) $AgNO_3$, white ppt. Blackens on heating. (N.B. Sulphites usually contain sulphates.)
„ „ SO_2 with deposition of sulphur	THIOSULPHATE	Same as for Sulphite. Distinguished by deposition of yellow sulphur on adding HCl and heating.
„ „ HCy (detected by odour, or by absorbing evolved gas on filter paper spotted with KHO, and applying confirmatory test to the spot) . . . . Repeat test for sulphide, using *strong* HCl, since some sulphides are not decomposed by the dilute acid .	CYANIDE	(1)‖ To neutral or alkaline solution, add $FeSO_4$, Fe_2Cl_6, and HCl; dark blue ppt. or colouration. (Prussian blue.) (2) $AgNO_3$, white ppt. soluble in NH_3, insoluble in dil. HNO_3. (N.B. Cyanides often contain cyanates and carbonates.)
2. Heat solid substance¶ with concentrated *Sulphuric Acid.*		
The above salts give much the same result as with hydrochloric acid. In addition to which		
Evolution of ClO_2 (yellow explosive gas) and liquid turning yellow	CHLORATE	(1) Indigo bleached in the *cold*, on the addition of *sulphurous acid.* (2) Heated, they evolve O, becoming chlorides.

For explanation of the changes which occur, see next page

and also special reactions of the respective Acid Radicles.

„	„ Oxides of nitrogen (yellow or brown, with characteristic odour)	NITRATE or	(1) Add $FeSO_4$ in excess to *cold* solution; gradually pour in strong H_2SO_4. Brown or pink ring where liquids meet. (2) Brucine in presence of strong H_2SO_4. Red colour.
		Nitrite	(1) KI + dilute HCl. Iodine liberated. Detected by starch. (2) $FeSO_4$ brown colour, with or without H_2SO_4.
„	„ Acetic acid vapour (recognized by odour).	ACETATE	(1) Fe_2Cl_6, red colour, bleached by HCl, not by $HgCl_2$. This test must be applied in *neutral* solution. (2) Heated with strong H_2SO_4 and a drop of alcohol, odour of ethyl acetate.
„	„ CO (burns with blue flame) and CO_2 (detected by lime-water as above) without blackening	OXALATE (Ferricyanide)	Proceed to Table on page 72.
„	„ CO alone, *without blackening*	FERROCYANIDE Cyanide Formate	
„	„ CO *with blackening* of the liquid . . .	TARTRATE (blackens at once) or CITRATE (blackens slowly)	
„	„ I (violet fumes, which colour starch paste blue)	IODIDE	
„	„ Br (brownish-red vapours, condensing to deep red liquid)	BROMIDE Bromate or Hypobromite	
„	„ SiF_4 (white clouds which deposit white gelatinous silica on a wet rod)	FLUORIDE or SILICOFLUORIDE	
„	„ HCl (colourless pungent fumes, forming white clouds in the air)	CHLORIDE	

If none of these results with the above reagents, the salt may be a phosphate, arsenate, borate, chromate, sulphate, silicate. Proceed to Table on page 72.

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[*] Cyanates treated with dilute acids evolve CO_2, and form an ammonium salt. The latter may be confirmed by adding caustic potash till alkaline, and Nessler's solution, when a yellow or brown colouration is obtained.

[†] Chlorates may also evolve Cl mixed with ClO_2.

[‡] Sulphocyanates give H_2S and CO_2. Distinguished by wet tests.

[§] Strong HCl vapours will also turn pot bichromate green after a time, and the odour of HCl is sometimes mistaken for that of SO_2; confirmatory test is therefore absolutely necessary.

[‖] Mercuric cyanide must be decomposed by H_2S before applying confirmatory test for cyanide.

[¶] If the substance given is a liquid, it should be evaporated *just* to dryness (with addition of a little caustic potash if acid). In many cases a strong solution may be used with excess of sulphuric acid; but the effects are not so well marked.

EXPLANATION OF PRELIMINARY ACID TABLE.

The following equations may be taken as typical of the reactions which occur:

Carbonate ......$Na_2CO_3 + 2HCl = 2NaCl + H_2O + \mathbf{CO_2}$.

Hypochlorite ...$\left\{\begin{array}{l} NaClO + HCl = NaCl + HClO \\ HClO + HCl = H_2O + \mathbf{Cl_2} \end{array}\right.$

Sulphide ....$Na_2S + 2HCl = 2NaCl + \mathbf{H_2S}$.

Sulphite ....$Na_2SO_3 + 2HCl = 2NaCl + H_2O + \mathbf{SO_2}$.

Thiosulphate ...$Na_2S_2O_3 + 2HCl = 2NaCl + H_2O + \mathbf{SO_2} + \mathbf{S}$.

Cyanide ....$KCy + HCl = KCl + HCy$.

Cyanate ....$\left\{\begin{array}{l} KCNO + HCl = HCNO + KCl \\ HCNO + H_2O + HCl = \mathbf{NH_4Cl} + \mathbf{CO_2} \end{array}\right.$

Chlorate ....$3KClO_3 + 2H_2SO_4 = 2KHSO_4 + KClO_4 + H_2O + \mathbf{2ClO_2}$.

Nitrate ....$\left\{\begin{array}{l} KNO_3 + H_2SO_4 = KHSO_4 + HNO_3 \\ 4HNO_3 \text{ (in part)} = 2H_2O + \mathbf{4NO_2} + O_2 \end{array}\right.$

Nitrite ....$2KNO_2 + 2H_2SO_4 = 2KHSO_4 + \mathbf{N_2O_3}$ (?) + H_2O (in part).

Acetate ....$CH_3COOK + H_2SO_4 = KHSO_4 + \mathbf{CH_3COOH}$.

Oxalate ....$\left\{\begin{array}{l} COOH \\ COOH \end{array}\right. - H_2O = \mathbf{CO} + \mathbf{CO_2}$.

Ferrocyanide ...$K_4FeC_6N_6 + 6H_2SO_4 + 6H_2O = 2K_2SO_4 + FeSO_4 + 3(NH_4)_2SO_4 + \mathbf{6CO}$.

Formate ....$HCOOH - H_2O = \mathbf{CO}$.

Cyanide ....$2KCN + 4H_2SO_4 + 2H_2O = 2KHSO_4 + 2(NH_4)HSO_4 + \mathbf{2CO}$ (in part).

Iodide ....$\left\{\begin{array}{l} KI + H_2SO_4 = KHSO_4 + HI \\ 2HI + H_2SO_4 = 2H_2O + SO_2 + \mathbf{I_2} \text{ (in part)*} \end{array}\right.$

Bromide ....Same reaction as iodide, but much HBr escapes undecomposed.

Bromate ....$\left\{\begin{array}{l} KBrO_3 + H_2SO_4 = KHSO_4 + HBrO_3 \\ 4HBrO_3 - 2H_2O = \mathbf{2Br_2} + 5O_2 \text{ (in part)} \end{array}\right.$

Hypobromite ...Same reaction as for hypochlorite.

Fluoride ....$\left\{\begin{array}{l} CaF_2 + H_2SO_4 = CaSO_4 + 2HF \\ 4HF + SiO_2 = 2H_2O + \mathbf{SiF_4} \end{array}\right.$

Silicofluoride ...$K_2SiF_6 + H_2SO_4 = K_2SO_4 + \mathbf{SiF_4} + 2HF$.

Chloride ....$NaCl + H_2SO_4 = NaHSO_4 + \mathbf{HCl}$.

$\left.\begin{array}{l} \text{Tartrate ....} \\ \text{Citrate ....} \end{array}\right\}$ Reactions not definite.

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* If the HI were much in excess of the H_2SO_4, some H_2S might be evolved—

$$8HI + H_2SO_4 = H_2S + 4H_2O + 4I_2.$$

EXAMINATION FOR ACID RADICLES IN THE WET WAY.

Preparation of Solution.

The substance, if solid, must be dissolved if possible in water, since acids are obviously inadmissible. [Dilute nitric acid may however be used in special cases, as for some acids of Group 3.]

If *insoluble in water*, the most *general* method is to boil with a strong solution of sodium carbonate for some time (or, in case of acids not decomposable by heat, to fuse with solid sodium carbonate and boil residue with water) and filter. In this way the acid is usually obtained as a soluble sodium salt, while the base remains as an insoluble carbonate or oxide, *e.g.*

$$CaF_2 + Na_2CO_3 = \underbrace{CaCO_3}_{\text{residue}} + \underbrace{2NaF.}_{\text{solution}}$$

If metals are present in solution which would interfere with the tests about to be applied (as a rule, all except K, Na or (NH_4)), they must be removed before proceeding. [The necessity, or otherwise, of removing metals, is roughly indicated by boiling a portion of the solution with Na_2CO_3; if no ppt. is produced, it is *usually* safe to proceed. The previous examination for metals, however, is a better guide.] This may in most cases be done by boiling or fusing with Na_2CO_3 as above. [Phosphates of Al, Ca, Ba, &c. are very imperfectly decomposed in this way, but they will have been identified during the examination for metals.]

The solution, after boiling or fusing with Na_2CO_3, must be just acidified with dilute HNO_3 to remove the excess of Na_2CO_3, and gently heated to expel CO_2.

Hydrosulphuric acid may be used to remove metals of groups 1 and 2, the filtrate being afterwards heated to expel excess of H_2S. This method is advisable in the cases of mercuric cyanide and tartar-emetic.

Dilute sulphuric acid and alcohol is sometimes employed in case of Pb, Ba, Sr and Ca.

EXAMINATION FOR ONE ACID RADICLE.

[The presence of Carbonate, Hypochlorite, Sulphide, Sulphite, and Thiosulphate, must be concluded from Preliminary Examination on page 68.]

Dissolve substance if possible in water. [If solution is alkaline, just acidify with dil. HNO_3 and filter off any ppt.†]

Test a portion of the solution by boiling with Na_2CO_3.

If the substance is ***insoluble in water***, or if the solution ***gives a ppt.*** with Na_2CO_3, boil* with strong solution of Na_2CO_3 for some time. Filter. Add dilute HNO_3 to the solution till just acid†. Heat gently to expel CO_2.

Test separate portions of the solution as follows:

1.	**Add dilute HCl + *Barium Chloride*. White ppt. indicates**		**CONFIRMATORY TESTS**
	SULPHATE	.	$Sr(NO_3)_2$, white ppt. (slowly in dilute solutions).
	SILICOFLUORIDE		**Heated alone, or with H_2SO_4, in platinum vessel, SiF_4 evolved, detected by wet rod.**
2.	**Add NH_3 till alkaline (filter if necessary†) and *Calcium Chloride*. White ppt. indicates**	**Digest ppt. with acetic acid**	
	OXALATE	**Undissolved**	**Treated with strong H_2SO_4 and MnO_2, CO_2 is evolved without heating.**
	FLUORIDE	,,	**(1) Heated in platinum vessel with strong H_2SO_4. HF is evolved; detected by etching glass.** (2) Heated in a test-tube with strong H_2SO_4, SiF_4 is evolved; detected by wet rod.
	PHOSPHATE	**Dissolved**	**(1)** In neutral solution, $AgNO_3$, yellow ppt. (2) **In NH_3 solution,** $MgSO_4$ **+ NH_4Cl, white ppt.** (3) In HNO_3 solution, $(NH_4)_2MoO_4$, **yellow ppt. on heating. (4) In acetic acid solution, Fe_2Cl_6, yellowish ppt.**
	ARSENATE	,,	(1) In neutral solution, $AgNO_3$, brick-red ppt. (2) H_2S in presence of HCl, yellow ppt. **on heating. (3) $MgSO_4$, Fe_2Cl_6, and $(NH_4)_2MoO_4$, same as with Phosphate.**
	BORATE	,,	**(1) Add concentrated H_2SO_4 and alcohol. Set fire to the alcohol. Green-edged flame.** **(2) Acidulate with dilute HCl. Dip turmeric paper, and dry it at gentle heat. Orange colour, changed to black or green by KHO.**
	TARTRATE	‡,,	**(1) Potassium acetate + acetic acid. white crystalline ppt. on shaking (in strong solution). (2) Add KHO *in excess* and a few drops of $K_2Mn_2O_8$. Green colour, changing to brown ppt. on heating. (3) Mirror test.**
	CITRATE §	,,	**(1) To neutral or slightly acid solution, add lime-water in excess. White ppt. on *boiling only*. (2) Add KHO *in excess* and a few drops of $K_2Mn_2O_8$. Green colour, not changed on heating.**
	(SILICATE)	**Decomposed; often with separation of silica**	**Evaporate HCl solution to dryness and heat; residue insoluble in HCl.**

	Add *Silver Nitrate* + dilute nitric acid. Ppt. indicates	Digest ppt. with ammonia	
3.	CHLORIDE	Easily soluble	(1) Heat with MnO_2 and strong H_2SO_4. Cl is evolved; detected by odour, and action on litmus, KI, &c. (2) Heat solid substance with dry $K_2Cr_2O_7$ and concentrated H_2SO_4. CrO_2Cl_2 is evolved (red gas); which when passed into water gives chromic acid (detected as below) and HCl.
	CYANIDE	”	Prussian blue test. See Preliminary Table.
	SULPHOCYANATE (white)	”	Fe_2Cl_6, blood-red colouration; bleached by $HgCl_2$, not by dilute HCl.
	FERRICYANIDE (orange)	”	(1) $FeSO_4$, dark blue ppt. (2) KI + dilute HCl, Iodine is liberated on heating. (3) Fe_2Cl_6, no ppt.
	BROMIDE (yellowish white)	Sparingly soluble	(1) Add a few drops of chlorine water, and shake up with CS_2; the latter is coloured yellow or orange. (2) Heated with strong H_2SO_4 and MnO_2, Bromine is evolved (orange-red gas).
	IODIDE (yellow)	Insoluble	(1) Add a few drops of chlorine water, and shake up with CS_2; the latter is coloured violet. (2) Heated with strong H_2SO_4 and MnO_2, Iodine is evolved (violet fumes; colour starch-paste blue).
	FERROCYANIDE (white)	”	(1) Fe_2Cl_6, dark blue ppt. (2) $CuSO_4$, chocolate ppt. (3) $FeSO_4$, pale blue ppt.
	SULPHIDE (black)	*”	See Preliminary Table.
	CHROMATE (crimson) In strong solution only	Soluble	See 5.

	Add *Ferric chloride* to neutral solution.		
4.	Blood-red colour indicates	Sulphocyanate	Bleached by $HgCl_2$; not by dilute HCl.
		Acetate	Not bleached by $HgCl_2$; bleached by HCl.
		(Iodide)	Red or brown ppt. with $HgCl_2$. Violet colour with CS_2.
	Yellowish white ppt. indicates	Phosphate Arsenate	See above.

5. If no result with the above reagents, test specially for CHROMATE.
(1) Lead acetate yellow ppt. insoluble in acetic acid.
(2) Boil with alcohol + HCl. The yellow or orange colour is changed to green.
Also for NITRATE and CHLORATE, since these may have been overlooked. See Preliminary Table.

6. If no acid radicle be found, the substance may be a metallic oxide or hydroxide. These, if soluble in water, give a brown ppt. with silver nitrate soluble in NH_3 or acids, and are alkaline to test-paper. If insoluble, the general properties of the body must be considered and compared with those of the oxide of the metal which has been found.

---

* Or fuse with excess of dry Na_2CO_3, extract fused mass with water and filter. This is preferable in many cases (minerals, &c.) but is inapplicable with organic acids and others decomposable by heat.

† A ppt. on neutralization is probably SiO_2 or Al_2O_3.

‡ Calcium tartrate often becomes crystalline, especially after a time, and will not then dissolve in acetic acid.

§ Citrate often gives no ppt. with $CaCl_2$ till boiled.

DETECTION OF MORE THAN ONE ACID RADICLE.

Preliminary examination as before. Make solution and remove bases if necessary, as directed on page 71.
Divide solution into several parts and test as below.

1

Acidulate with dilute HCl, and add Barium Chloride—

White ppt. indicates SULPHATE, SILICOFLUORIDE.

- If Silicofluoride is suspected (Preliminary Table), confirm by heating this ppt. with concentrated H_2SO_4 in a platinum vessel.
- Confirm Sulphate in another portion of solution by adding HCl (dil.) and strontium nitrate.

2

Add NH_3 till alkaline (filter off any ppt.*) and Calcium Chloride—

White ppt. indicates *Oxalate, Fluoride, Phosphate, Arsenite, Borate, Tartrate, Citrate.* (Sulphate, Carbonate, Silicate.)

Filter†, wash ppt. and digest it with acetic acid.

FILTER

RESIDUE. *Oxalate, Fluoride*‡ (Sulphate)	SOLUTION
Wash; heat to low redness and treat with acetic acid. Effervescence indicates OXALATE.	Add Fe_2Cl_6 drop by drop.
Examine residue insoluble in acetic acid for FLUORIDE.	Yellowish-white ppt. indicates *Phosphate*, *Arsenate*
	Dissolve ppt. in dilute HCl, boil, pass H_2S for some time, and again boil. Yellow ppt. indicates ARSENATE. If arsenate is present, it must be entirely removed by H_2S before testing for PHOSPHATE by ammonium molybdate.

3

Add Silver Nitrate and dilute HNO_3—

Ppt. indicates	*Chloride*	*Cyanide*	*Sulphocyanate*	*Ferricyanide*	*Bromide*	*Iodide*	*Ferrocyanide*	(Sulphide)
	(white)	(white)	(white)	(orange)	(yellowish-white)	(yellow)	(white)	(black)
	Easily soluble in NH_3 (A)				Sparingly soluble in NH_3 (B)	Insoluble in NH_3 (C)		

Digest ppt. with excess of NH_3 for some time. Filter. Residue = (C). Acidulate filtrate with HNO_3. Ppt. = (A) + (B).

4§

Acidulate with dilute HCl, and to a portion add Ferric Chloride.

Dark blue ppt. FERROCYANIDE. Blood-red colouration bleached by $HgCl_2$ SULPHOCYANATE.

If no effect, to another portion add Ferrous Sulphate. Dark blue ppt. FERRICYANIDE.

5

Test specially for BORATE by adding concentrated H_2SO_4 and alcohol, and setting fire to the mixture. Also by turmeric test.
If blackening occurred in the preliminary examination, test specially for TARTRATE and CITRATE as below.

Test specially also for ACETATE, CHROMATE, NITRATE and CHLORATE, as in detecting one acid radicle (or as below).

Results obtained with the above tests will indicate which, if any, of the following separations are necessary:

NITRATE and NITRITE.

To one portion add KI + dilute HCl. Iodine liberated indicates *nitrite*.

$$HNO_2+HI=H_2O+NO+I.$$

If nitrite is present, to another portion add urea + dilute H_2SO_4 and heat; nitrite is thus destroyed,

$$2HNO_2+CO(NH_2)_2=CO_2+3H_2O+2N_2.$$

Test residual solution for *nitrate* as usual with $FeSO_4$ and concentrated H_2SO_4.

NITRATE and CHLORATE

Heat solid to redness. Nitrate becomes nitrite, chlorate becomes chloride,

$$KNO_3=KNO_2+O$$
$$KClO_3=KCl\ +3O.$$

Dissolve residue in water; test one portion for nitrite by KI and dilute HCl as above, and another for chloride by $AgNO_3+HNO_3$.

SULPHITE and CARBONATE.

Add HCl. SO_2 and CO_2 are evolved. Pass the gases through a solution of $K_2Cr_2O_7$ acidulated with H_2SO_4. SO_2 is absorbed, and the solution turns green.

$$K_2Cr_2O_7+H_2SO_4+3SO_2=K_2SO_4+Cr_2(SO_4)_3+H_2O.$$

Test residual gas for CO_2 by lime-water.

SULPHATE, SULPHITE and THIOSULPHATE.

To a dilute neutral solution add $BaCl_2$ in excess. Filter.

Ppt.	Solution
Sulphate, Sulphite. Wash. Digest with dilute HCl. Filter.	Add HCl and boil; yellow ppt. of S and odour of SO_2 indicates *Thiosulphite* $H_2S_2O_3=H_2O+SO_2+S$.

Residue	Solution
Sulphate	Add chlorine water. White ppt. indicates *Sulphite* $BaSO_3+Cl_2+H_2O=BaSO_4+2HCl$.

CHLORIDE, BROMIDE and IODIDE.

To neutral solution add $CuSO_4$ in excess, and H_2SO_3, cuprous iodide is precipitated,

$$2KI+2CuSO_4+H_2SO_3+H_2O=Cu_2I_2+2KHSO_4+H_2SO_4.$$

Filter, confirm *iodide* in ppt. by heating with concentrated H_2SO_4. To filtrate, add KHO in excess and boil, to remove excess of Cu as CuO. Filter, evaporate solution to dryness. Mix dry residue with dry $K_2Cr_2O_7$ and concentrated H_2SO_4. Distil. Bromine and chromyl dichloride are evolved,

$$6KBr+K_2Cr_2O_7+7H_2SO_4=4K_2SO_4+Cr_2(SO_4)_3+7H_2O+3Br_2$$
$$4KCl+K_2Cr_2O_7+3H_2SO_4=3K_2SO_4+2CrO_2Cl_2+3H_2O.$$

Pass the gases into water. Br dissolves. CrO_2Cl_2 is decomposed thus,

$$CrO_2Cl_2+2H_2O=H_2CrO_4+2HCl.$$

Confirm *Bromine* by shaking up with CS_2; and chromate (and consequently *chloride*) by neutralizing, adding acetic acid and lead acetate

IODIDE and IODATE.

Confirm iodide in one portion by adding chlorine water + CS_2. If iodide is present, add dilute HCl (+ starch or CS_2) to another portion. Iodine liberated indicates *Iodate*.

$$5HI+HIO_3=3H_2O+3I_2.$$

FERROCYANIDE, FERRICYANIDE and SULPHOCYANATE ||.

To a portion acidulated with dilute HCl add Fe_2Cl_6 in excess. Filter.

Ppt.	Solution
Wash with $HgCl_2$; if dark blue, *Ferrocyanide.*	Blood-red bleached by $HgCl_2$, *Sulphocyanate.*

To another portion add KI + dilute HCl and heat. Iodine liberated (detected by shaking with CS_2) indicates *Ferricyanide*,

$$H_6Fe_2Cy_{12}+2HI=2H_4FeCy_6+I_2.$$

TARTRATE and CITRATE.

To a *concentrated* solution add excess of potassium acetate dissolved in alcohol, and if necessary, a few drops of acetic acid. Add an equal bulk of alcohol, shake, stir, and allow to stand for some time. Filter.

Ppt.	Solution
(crystalline, often adhering to the sides of vessel) indicates *Tartrate.* Dissolve it in least possible KHO, and confirm by "mirror" test (or $FeSO_4+H_2O_2$ &c. test).	Confirm citrate by the $CaCl_2$ test after boiling off alcohol and neutralizing with NH_3. Or by Barium acetate after neutralizing.

---

* Probably SiO_2 or Al_2O_3. † A ppt. on boiling filtrate is probably *Citrate.* ‡ See note ‡, page 78. § Unnecessary if no ppt. in (3).

|| Cyanide may be detected in presence of Ferrocyanide, &c. by distilling with solution of potassium hydrogen tartrate, which causes HCy to be evolved from cyanide only.

EXAMINATION FOR ACID RADICLES.

Explanation.

The method of examination here recommended depends upon the following principles:

1. The insolubility of Sulphate and Silicofluoride of Barium in dilute HCl.

   These two acid radicles are easily distinguished in presence of one another, since silicofluorides heated with concentrated H_2SO_4 evolve SiF_4 and HF,

   $$BaSiF_6 + H_2SO_4 = BaSO_4 + SiF_4 + 2HF,$$

   whilst sulphates give a white ppt. of $SrSO_4$ with a strontium salt.

2. The insolubility (or sparing solubility) of the following calcium salts in water. Oxalate, Fluoride, Phosphate, Arsenate, Borate, Tartrate, Citrate, (Carbonate).

   Of these, calcium oxalate and fluoride are insoluble, or nearly so, in acetic acid, the rest being soluble.

   Calcium oxalate on heating to redness is converted into carbonate,

   $$Ca(CO_2)_2 = CaCO_3 + CO,$$

   and the residue after heating therefore effervesces when treated with a dilute acid. Calcium fluoride remains unaltered.

   From the solution of the remaining calcium salts in acetic acid, ferric chloride precipitates phosphate and arsenate,

   $$Ca_3(PO_4)_2 + Fe_2Cl_6 = 2FePO_4 + 3CaCl_2.$$

   Tartrate, citrate, and borate are looked for in separate portions, the two former however only if indicated in the preliminary examination.

3. The insolubility of the following silver salts in dilute nitric acid. Chloride, Cyanide, Sulphocyanate, Ferricyanide, Bromide, Ferrocyanide, Iodide, (Sulphide). (If silver nitrate were added in *neutral* solution, nearly all other acid radicles would likewise be precipitated as silver salts.)

   These can be further subdivided into groups owing to their different solubilities in ammonia.

4. Ferric chloride, in dilute acid or neutral solution, precipitates ferrocyanide as "Prussian blue" (usually represented as $Fe_4(FeCy_6)_3$) and gives a blood-red colour with sulphocyanate due to ferric sulphocyanate $Fe_2(CyS)_6$; whilst ferrous sulphate precipitates ferricyanide as "Turnbull's blue" $Fe_3(Fe_2Cy_{12})$. If ferrocyanide is present, ferricyanide must be looked for in some other way, such as by adding potassium iodide and starch (see Special Separations), (or by the solubility of its silver salt in ammonia, but this is not satisfactory, since there is danger of reduction to ferrocyanide).

Having so far divided the acids which are present into groups, special tests or separations must be applied as the results obtained dictate. By carefully noting the effect of each test applied, and *excluding* those acid radicles shewn to be absent, there will usually remain but few which require further separation or confirmation. The identification of one acid radicle will, moreover, often indicate the presence or absence of another. For example, if a hypochlorite is found, a chloride is almost certain to be present (since pure hypochlorites are very rare and unstable; so-called hypochlorites being usually mixtures of hypochlorite and chloride), whereas sulphite must necessarily be absent, if in solution (since sulphite + hypochlorite = sulphate + chloride).

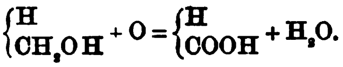
REACTIONS OF SOME OF THE MORE COMMON ORGANIC BODIES.

METHYL ALCOHOL.

Wood Spirit. $CH_3.OH$

Colourless, limpid, volatile, inflammable liquid. Boils at 66° C. Miscible with water, ether, and ethyl alcohol.

Heated with *potassium dichromate and dilute sulphuric acid* in *excess*, it yields formic acid, which may be distilled off and identified as usual,



$$\left\{\begin{matrix}H\\CH_2OH\end{matrix}\right. + O = \left\{\begin{matrix}H\\COOH\end{matrix}\right. + H_2O.$$

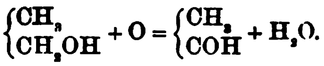
Iodine and caustic potash yields no iodoform.

ETHYL ALCOHOL.

Alcohol. Spirits of Wine. $(C_2H_5)OH$ or $\left\{\begin{matrix}CH_3\\CH_2OH\end{matrix}\right.$.

Colourless volatile inflammable liquid. Boils at 78°.4 C. Miscible with water and with ether.

1. Heated with *potassium dichromate and dilute sulphuric acid,* not in excess, it yields acetaldehyde—



$$\left\{\begin{matrix}CH_3\\CH_2OH\end{matrix}\right. + O = \left\{\begin{matrix}CH_3\\COH\end{matrix}\right. + H_2O.$$

The aldehyde may be distilled off and identified by the usual tests.

2. A dilute solution of ethyl alcohol gently heated, and a colourless strong solution of *iodine* in *caustic potash* added, gives gradually a yellow crystalline ppt. of iodoform CHI_3. Examined under the microscope, this ppt. is seen to consist of hexagonal plates or six-rayed stars. [Lieben's test.]*
3. Heated with *sodium acetate* and strong *sulphuric acid*, the characteristic odour of ethyl acetate (acetic ether) is produced,

$$(C_2H_5)OH + CH_3.COOH = CH_3.COO(C_2H_5) + H_2O.$$

---

* This reaction is also produced by aldehyde, acetone, ethylidene-lactic acid, &c. but not by chloroform, chloral, methyl or amyl alcohol, acetic or formic acids, glycerin or ether.

ETHYL ETHER.

Ordinary Ether. "Sulphuric" Ether. $(C_2H_5)_2O$.

Colourless, very mobile liquid. Penetrating odour. Highly volatile, its rapid evaporation causing great reduction of temperature. Boils at 35° C. Highly inflammable. Miscible in all proportions with alcohol, but only to a limited extent with water. Dissolves various fats, oils, resins, &c.

By slow oxidation, *e. g.* with air under certain conditions, it yields aldehyde, acetic acid, hydrogen dioxide, &c.

Heated with nitric acid it gives acetic acid, oxalic acid and carbon dioxide.

ACETALDEHYDE.

Aldehyde. Ethaldehyde. $\left\{\begin{matrix}CH_3\\COH\end{matrix}\right.$.

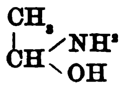
Colourless mobile inflammable liquid. Boils at 20°. 8 C. Characteristic odour. Miscible with water, alcohol, and ether.

1. Solution of *silver oxide* in ammonia gives a bright mirror of metallic silver on warming. (More delicate in presence of KHO.)

2. *Fehling's solution* gives, on heating, a red ppt. of Cu_2O.
   In these cases the aldehyde is converted into acetic acid,

$$\left\{\begin{matrix}CH_3\\COH\end{matrix}\right. + O = \left\{\begin{matrix}CH_3\\COOH\end{matrix}\right. .$$

3 Heated with *caustic potash* the liquid turns brown, owing to the formation of so-called aldehyde-resin.

4. If dissolved in ether, and *ammonia* gas be passed into the liquid, white crystals of "ammonia-aldehyde"  $\begin{matrix}CH_3\\ |\\ CH\begin{matrix}\diagup NH_2\\ \diagdown OH\end{matrix}\end{matrix}$ are obtained.

5. A solution of a *rosaniline salt*, which has been bleached by sulphur dioxide, gives a violet or pink colour.

CHLORAL.

Trichlor-acetaldehyde. $\left\{\begin{matrix}CCl_3\\ COH\end{matrix}\right.$.

Colourless mobile liquid. Boils at 97°. 2 C. Combines readily with water, forming crystalline chloral hydrate,

$$\left\{\begin{matrix}CCl_3\\ COH\end{matrix}\right. . H_2O \text{ or } \left\{\begin{matrix}CCl_3\\ CH(OH)_2\end{matrix}\right. .$$

1. *Caustic potash* (aqueous solution) converts it into chloroform and potassium formate,

$$\left\{\begin{matrix}CCl_3\\ COH\end{matrix}\right. + KHO = CHCl_3 + \left\{\begin{matrix}H\\ COOK\end{matrix}\right. .$$

These products may be identified as usual.

2. *Silver oxide, Fehling's solution*, and *ammonia-gas*, behave as with acetaldehyde.

CHLOROFORM.

Trichloromethane. $CHCl_3$.

Colourless limpid volatile liquid. Boils at 61°.2 C. Soluble in alcohol and in ether; nearly insoluble in water.

1. Heated with an alcoholic solution of *caustic potash*, it gives potassium formate and potassium chloride,

$$CHCl_3 + 4KHO = \left\{\begin{matrix}H\\ COOK\end{matrix}\right. + 3KCl + 2H_2O.$$

These products may be identified as usual.

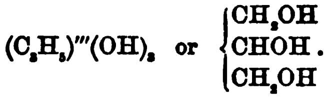
2. Heated with a *primary amine* (*e.g.* aniline) and an alcoholic solution of caustic potash, the characteristic odour of carbamines is produced,

$$CHCl_3 + \underset{\text{aniline}}{(C_6H_5)NH_2} + 3KHO = \underset{\text{Phenyl carbamine}}{(C_6H_5)NC} + 3KCl + 3H_2O.$$

3. *Fehling's solution* is readily reduced on heating, red Cu_2O being precipitated,

$$CHCl_3 + 2CuO + 5KHO = Cu_2O + K_2CO_3 + 3KCl + 3H_2O,$$

GLYCERIN.



$$(C_3H_5)'''(OH)_3 \text{ or } \left\{\begin{matrix} CH_2OH \\ CHOH \\ CH_2OH \end{matrix}\right. .$$

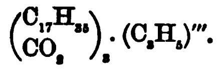
Colourless viscid liquid. Sweet taste. Miscible with water and with alcohol.

1. Heated with *dehydrating agents* (*e.g.* $KHSO_4$ or concentrated H_2SO_4), characteristic irritating odour of acrolein is obtained,

$$C_3H_5(OH)_3 - 2H_2O = C_2H_3 . COH.$$

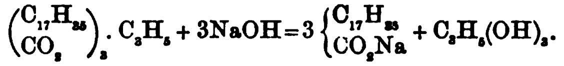
2. A *borax bead* immersed for a few minutes in a solution of glycerin (made slightly alkaline with KHO) and held in a Bunsen flame, gives a green colour to the flame.

FATS.

E.g. **Tri-stearin (Mutton fat)**  $\begin{pmatrix} C_{17}H_{35} \\ CO_2 \end{pmatrix}_3 . (C_3H_5)'''.$

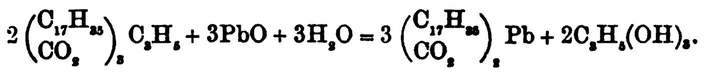
Soluble in ether, and in carbon disulphide. Practically insoluble in water.

1. Heated with *caustic soda* or *potash**, a soap is produced and glycerin separates,



$$\begin{pmatrix} C_{17}H_{35} \\ CO_2 \end{pmatrix}_3 . C_3H_5 + 3NaOH = 3\left\{\begin{matrix} C_{17}H_{35} \\ CO_2Na \end{matrix}\right. + C_3H_5(OH)_3.$$

2. And with *lead oxide*, insoluble lead stearate and glycerin are produced,



$$2\begin{pmatrix} C_{17}H_{35} \\ CO_2 \end{pmatrix}_3 C_3H_5 + 3PbO + 3H_2O = 3\begin{pmatrix} C_{17}H_{35} \\ CO_2 \end{pmatrix}_2 Pb + 2C_3H_5(OH)_3.$$

3. Heated alone they decompose, evolving acrolein $C_2H_3 . COH$, and other offensive vapours.

---

* Best in alcoholic solution.

SOAPS.

E.g. **Sodium Stearate.** $\left\{\begin{matrix}C_{17}H_{35}\\COONa\end{matrix}\right.$.

Soluble in pure water; reprecipitated by a strong solution of sodium chloride. Soluble in alcohol. Aqueous solution "lathers" when shaken.

1. From alcoholic or aqueous solution, *calcium* or *magnesium salts* (*e.g.* hard water) precipitate white calcium or magnesium stearates,

$$CaSO_4 + 2\left\{\begin{matrix}C_{17}H_{35}\\CO_2Na\end{matrix}\right. = \begin{pmatrix}C_{17}H_{35}\\CO_2\end{pmatrix}_2 Ca + Na_2SO_4.$$

2. Dilute *hydrochloric acid* gives a white ppt. of stearic acid,

$$\left\{\begin{matrix}C_{17}H_{35}\\COONa\end{matrix}\right. + HCl = \left\{\begin{matrix}C_{17}H_{35}\\COOH\end{matrix}\right. + NaCl.$$

CARBOHYDRATES.

I. Saccharons. ($C_{12}H_{22}O_{11}$.)

Cane Sugar. Saccharon (Sucrose). $C_{12}H_{22}O_{11}$.

Soluble in water. Insoluble in cold absolute alcohol.
Dextrorotatory.

1. Does not directly reduce *Fehling's solution**.

2. Boiled with *dilute acids* (H_2SO_4 or HCl) it takes up the elements of water, forming a mixture of dextrose and lævulose ("inverted sugar"),

$$C_{12}H_{22}O_{11} + H_2O = C_6H_{12}O_6 + C_6H_{12}O_6.$$

3. In contact with *yeast* it passes first into a mixture of dextrose and lævulose, which then undergo alcoholic fermentation.

4. *Concentrated sulphuric acid*, added to a strong warm solution, causes it to blacken and swell up, owing to the separation of carbonaceous matter, CO, SO_2, HCOOH, &c. being evolved.

---

* "Fehling's solution" is made from a cupric salt, an alkaline tartrate, and caustic alkali. The action of glucoses upon it, is virtually the reduction of CuO to Cu_2O, the glucose becoming oxidized to acetic, formic, tartronic, &c. acids, and other bodies. The exact nature of the change is not known.

CARBOHYDRATES (*continued*).

Milk Sugar. Lacton. $C_{12}H_{22}O_{11}$.

Less soluble in water than cane sugar. Insoluble in absolute alcohol.
Dextrorotatory.

1. Readily reduces *Fehling's solution* on heating, red Cu_2O being precipitated.
2. Boiled with *dilute acids*, it takes up the elements of water, forming a mixture of galactose ($C_6H_{12}O_6$), and another glucose ($C_6H_{12}O_6$), which is probably identical with sucrodextrose.
3. Not directly fermentable by yeast.
4. *Concentrated sulphuric acid* chars it, but less readily than cane sugar.

Starch Sugar. Malton (Maltose) Amylon. $C_{12}H_{22}O_{11}$.

Soluble in water and in alcohol.
Dextrorotatory. Diffusible.

1. Reduces *Fehling's solution* on heating, red Cu_2O being precipitated.
2. Boiled with *dilute acids*, it is slowly converted into a mixture of two dextroglucoses $C_6H_{12}O_6$.*
3. In contact with *yeast* it is first converted (probably) into dextroglucoses, which then undergo alcoholic fermentation.

II. Glucoses. ($C_6H_{12}O_6$.)

Dextrose. Sucrodextrose. Grape sugar. $C_6H_{12}O_6$.

Soluble in water and in alcohol.
Dextrorotatory.
Less sweet than cane sugar.

1. Reduces *Fehling's solution* at once on heating, red Cu_2O being precipitated.
2. A warm solution in contact with yeast readily undergoes alcoholic fermentation,
$$C_6H_{12}O_6 = 2(C_2H_5)OH + 2CO_2.$$
3. *Concentrated sulphuric acid* does not blacken it.
4. Heated with strong solution of *caustic potash* it turns brown.
5. Not affected by boiling with dilute H_2SO_4.

Lævulose. Sucrolævulose. $C_6H_{12}O_6$.

Resembles dextrose in the above properties, but is *lævorotatory*, sweeter, and more soluble in alcohol.

Galactose. Lactose. $C_6H_{12}O_6$.

A dextrorotatory glucose, closely resembling dextrose, but having a higher rotatory power, and non-fermentable by yeast.

---

* Or perhaps entirely into ordinary sucrodextrose. Diastase is said to have no action on amylon.

CARBOHYDRATES (*continued*).

III. Carbohydrates of the composition $(C_6H_{10}O_5)_n$.

Starch. $(C_6H_{10}O_5)_n$.

Insoluble in cold water. Heated with water it forms a mucilage or paste, and on boiling with much water, a kind of solution which contains "soluble starch".

Dextrorotatory. Colloid.

1. *Iodine* colours starch paste, or solution, blue. The colour is temporarily destroyed by heating.
2. Solid starch when heated to about 150° C. is converted into "British gum" (impure dextrin) which is soluble in cold water, and is not coloured blue by iodine.
3. By the action of many *unorganized ferments* (such as ptyalin, diastase, &c.) or by heating with *dilute acids*, starch takes up the elements of water, forming a mixture of dextrin $(C_6H_{10}O_5)_n$ and amylon $C_{12}H_{22}O_{11}$. These bodies afterwards undergo further hydration, giving dextroglucoses. (See Amylon and Dextrin.)

Dextrin. Amylin. $(C_6H_{10}O_5)_n$.

Easily soluble in cold water. Insoluble in alcohol.

Dextrorotatory. Colloid.

Solution is not coloured blue by *iodine**.

In contact with many unorganized ferments (or dilute acids), it is converted into amylon $C_{12}H_{22}O_{11}$,

$$2(C_6H_{10}O_5)_n + nH_2O = nC_{12}H_{22}O_{11}.$$

Inulin. $(C_6H_{10}O_5)_n$.

Resembles starch, but is *completely soluble* in warm water, the solution is *lævorotatory*, and is *not coloured blue* by *iodine*.

In contact with *dilute acids* it gives a lævorotatory glucose resembling sucrolævulose.

Glycogen. $(C_6H_{10}O_5)_n$.

Readily soluble in warm water, forming an opalescent solution. Insoluble in alcohol.

Dextrorotatory.

Iodine colours it reddish-brown. The colour is temporarily destroyed by heating.

By *dilute acids*, and by many unorganized ferments, it is converted first into a kind of dextrin (according to some, into a mixture of amylin and amylon) and then into a dextrorotatory glucose $C_6H_{12}O_6$, perhaps identical with sucrodextrose.

Cellulose. $(C_6H_{10}O_5)_n$.

Insoluble in water or alcohol. Soluble in a solution of cuprammonic hydroxide (prepared by dissolving cupric hydroxide in ammonia), from which it is reprecipitated by HCl or CO_2.

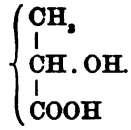
Iodine alone does not colour cellulose blue, but it does so in presence of concentrated sulphuric acid, or of zinc chloride.

---

* There are probably several varieties of dextrin. So-called erythro-dextrin gives a reddish-brown colour with iodine, which permanently disappears on heating.

LACTIC ACIDS.

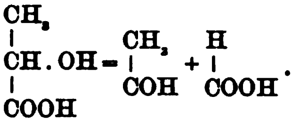
Fermentation Lactic Acid. (*Inactive* α Hydroxypropionic or Ethylidene Lactic Acid.)



$$\left\{\begin{array}{l}\mathrm{CH_3}\\ |\\ \mathrm{CH.OH.}\\ |\\ \mathrm{COOH}\end{array}\right.$$

Colourless syrupy liquid. Very soluble in water, alcohol, or ether.

1. Heated with *dilute sulphuric acid*, it yields acetaldehyde and formic acid,

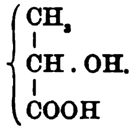


$$\begin{array}{l}\mathrm{CH_3}\\ |\\ \mathrm{CH.OH}\\ |\\ \mathrm{COOH}\end{array} = \begin{array}{l}\mathrm{CH_3}\\ |\\ \mathrm{COH}\end{array} + \begin{array}{l}\mathrm{H}\\ |\\ \mathrm{COOH}\end{array}.$$

2. *Gentle oxidizing agents* (*e.g.* manganese dioxide and dilute sulphuric acid) convert it into acetaldehyde and carbon dioxide. More powerful oxidizing agents yield acetic acid and carbon dioxide.

3. Heated with concentrated sulphuric acid, carbon monoxide is evolved.

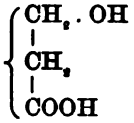
Sarcolactic Acid. (*Active* α Hydroxypropionic or Ethylidene Lactic Acid.)



$$\left\{\begin{array}{l}\mathrm{CH_3}\\ |\\ \mathrm{CH.OH.}\\ |\\ \mathrm{COOH}\end{array}\right.$$

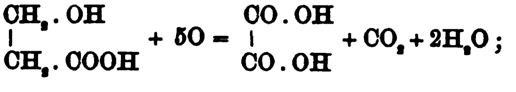
Identical with fermentation lactic acid in the above properties, but is distinguished by being *optically active* (feebly dextrorotatory). There is a difference also in the character of the zinc salts.

Ethylene Lactic Acid. (β Hydroxypropionic Acid.)



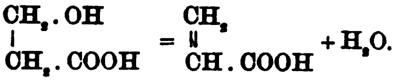
$$\left\{\begin{array}{l}\mathrm{CH_2.OH}\\ |\\ \mathrm{CH_2}\\ |\\ \mathrm{COOH}\end{array}\right.$$

Differs from the above in that, when treated with oxidizing agents (chromic or nitric acids), it yields *oxalic* acid and carbon dioxide—



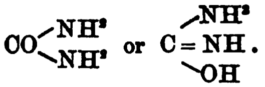
$$\begin{array}{l}\mathrm{CH_2.OH}\\ |\\ \mathrm{CH_2.COOH}\end{array} + 5\mathrm{O} = \begin{array}{l}\mathrm{CO.OH}\\ |\\ \mathrm{CO.OH}\end{array} + \mathrm{CO_2} + 2\mathrm{H_2O};$$

and when heated alone, or with dilute sulphuric acid, it splits up into acrylic acid and water—



$$\begin{array}{l}\mathrm{CH_2.OH}\\ |\\ \mathrm{CH_2.COOH}\end{array} = \begin{array}{l}\mathrm{CH_2}\\ \|\\ \mathrm{CH.COOH}\end{array} + \mathrm{H_2O}.$$

UREA.

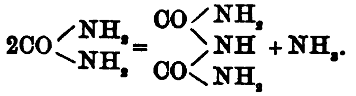
Carbamide (?) 

$$CO\begin{matrix}\diagup NH_2\\ \diagdown NH_2\end{matrix} \text{ or } C\begin{matrix}\diagup NH_2\\ = NH\\ \diagdown OH\end{matrix}.$$

Prismatic crystals. Easily soluble in water and in alcohol.

1. *Strong nitric acid* throws down, from concentrated solutions, crystals of urea nitrate $CO(NH_2)_2 . HNO_3$. Under the microscope these are seen to consist of rhombic or six-sided tables.

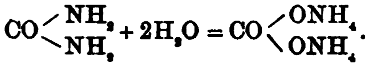
2. *Heated alone* to about 150° C. ammonia is evolved and biuret formed—



$$2CO\begin{matrix}\diagup NH_2\\ \diagdown NH_2\end{matrix} = \begin{matrix}CO\diagup NH_2\\ \quad\diagdown NH\\ CO\diagup NH_2\end{matrix} + NH_3.$$

On dissolving biuret in water, adding excess of caustic potash and a drop of *copper sulphate*, a red-violet colour is produced.

3. A neutral solution heated to about 40° C. with a trace of *urine-ferment*, turns alkaline after a few minutes, owing to formation of ammonium carbonate—



$$CO\begin{matrix}\diagup NH_2\\ \diagdown NH_2\end{matrix} + 2H_2O = CO\begin{matrix}\diagup ONH_4\\ \diagdown ONH_4\end{matrix}.$$

4. *Sodium hypobromite*, in presence of caustic soda*, causes a rapid evolution of nitrogen—

$$CO(NH_2)_2 + 3NaBrO + 2NaOH = 3NaBr + Na_2CO_3 + 3H_2O + N_2.$$

(Sodium *hypochlorite* only evolves half the nitrogen in the cold—

$$2CO(NH_2)_2 + 3NaClO + 2NaOH = 2NaCNO + 3NaCl + 5H_2O + N_2.)$$

5. Nitrous acid (or a nitrite + dilute acid) evolves nitrogen and carbon dioxide (complete on heating),

$$CO(NH_2)_2 + 2HNO_2 = CO_2 + 3H_2O + 2N_2.$$

6. Mercuric nitrate (in absence of chlorides), white ppt. of

$CO(NH_2)_2 . 2HgO$, or $2CO(NH_2)_2 . 3HgO . Hg(NO_3)_2$, according to conditions.

---

* Prepared by gradually adding bromine to strong caustic soda solution, and keeping the mixture cool.

URIC ACID.

$H_2(C_5H_2N_4O_3)$.

White crystalline powder. Almost insoluble in water. Soluble in caustic potash, forming potassium urate, $K_2(C_5H_2N_4O_3)$, from which hydrochloric acid reprecipitates uric acid.

Evaporated just to dryness with dilute *nitric acid*, a yellow or pinkish residue is left, which when moistened with *ammonia* gives a crimson colour, due (in part) to "murexide" or ammonium purpurate, $NH_4.(C_8H_4N_5O_6)$. This colour is changed to a purple by caustic potash.

Heated alone, it blackens, and evolves, amongst other bodies, hydrocyanic acid, carbon dioxide and ammonia, which may be recognized by their odour, &c.

STRYCHNINE.

Strychnia. $C_{21}H_{22}N_2O_2$.

Prismatic crystals or white powder. Very slightly soluble in water, the solution having a bitter taste. Sparingly soluble in alcohol and ether. Soluble in chloroform. Soluble in most acids, forming salts.

Dissolved in concentrated *sulphuric acid* and a fragment of *manganese dioxide** added, a purple colour is obtained, which rapidly changes to crimson, and afterwards to pink or orange.

[Curarine gives an exactly similar reaction, but it is almost insoluble in chloroform.] *Caustic potash* (ammonia, potassium, carbonate, &c.) throws down a white ppt. of strychnine from solutions of its salts (if not too dilute) which is insoluble in excess, and soon becomes crystalline.

QUININE.

Quinia. $C_{20}H_{24}N_2O_2$.

White powder or crystals. Very sparingly soluble in water. Easily soluble in alcohol and in chloroform; less so in ether. Soluble in most acids, forming salts.

1. Dissolved in *dilute sulphuric acid*, the solution exhibits well marked blue fluorescence.
2. A nearly neutral solution mixed with *chlorine or bromine water*, and then with *ammonia* in excess, gives a bright green colouration or precipitate.
3. Treated first with *chlorine or bromine water*, then with a few drops of *potassium ferro-* or *ferri- cyanide*, and lastly with *ammonia* not in excess, a red colouration is produced, destroyed by excess of ammonia.
4. *Caustic potash* gives, in solutions of quinine salts, in white ppt. of hydrated quinine, insoluble in excess.

---

* Potassium bichromate or permanganate, lead dioxide, &c. answer the same purpose.

MORPHINE.

Morphia. $C_{17}H_{19}NO_3 \cdot H_2O$.

White powder or small prisms. Almost insoluble in cold water; very sparingly soluble in boiling water, ether, cold alcohol, and chloroform. Dissolves easily in hot alcohol; also in dilute acids, forming salts.

1. Neutral *ferric chloride* gives with dry morphine (or a neutral solution) a deep inky blue colour, changed to green by excess of ferric chloride.
2. *Iodic acid* gives, in neutral morphine solutions, a brown colour, due in part to the liberation of iodine, which may be detected by a starch test. The brown colour is deepened by ammonia.
3. Strong *nitric acid* in excess, gives a deep orange colour. (Unchanged by stannous chloride.)
4. Heated to 100° C. with concentrated *sulphuric acid*, and a crystal of pure *potassium perchlorate* added, a brown colour is produced.
5. From strong solutions of morphine salts, *caustic potash* (one drop) gives, on stirring, a white ppt. of hydrated morphine, easily soluble in excess.

BRUCINE.

Brucia. $C_{23}H_{26}N_2O_4$.

White powder, or crystalline needles or prisms. Slightly soluble in water. Easily soluble in alcohol and in chloroform. Soluble in most acids, forming salts.

1. Strong *nitric acid* gives with solid brucine (or a solution to which strong sulphuric acid* has been added) a scarlet colouration changing to orange and yellow. *Stannous chloride* changes the colour to purple or violet, if sufficient brucine is present.
2. *Mercurous nitrate* added to a neutral solution, and the mixture heated to 100° gives a crimson colour.
3. Caustic potash precipitates brucine from solutions of its salts, insoluble in excess.

---

* Strong sulphuric acid itself gives a transient rose colour with brucine.

BENZENE.

Benzol. C_6H_6.

Colourless very limpid liquid. Characteristic odour. Gives off highly inflammable vapour when heated. Boils at 80°·5 C. Miscible with alcohol, ether, and chloroform, but not appreciably with water.

Agitated for a few minutes with the strongest *nitric acid* (not allowing the temperature to rise too high), nitrobenzene $C_6H_5NO_2$ is formed, which separates in oily drops on pouring the liquid into much water, and has characteristic odour of bitter almonds. On digesting nitrobenzene with a hydrogenizing agent, *e.g.* zinc and hydrochloric acid, aniline is obtained,

$$C_6H_5NO_2 + 3H_2 = C_6H_5NH_2 + 2H_2O,$$

which may be detected as usual.

PHENOL.

Carbolic Acid. Phenic Acid. $C_6H_5 . OH$.

Colourless deliquescent crystals. Characteristic odour. Sparingly soluble in water; solution does not redden litmus. Easily soluble in alcohol, ether, chloroform, or alkalies.

Aqueous solutions of phenol give with

1. *Bromine water*, white or yellowish ppt. of tribromophenol $C_6H_2Br_3 . OH$, even in very dilute solutions (1 in 60,000). This ppt. if washed, digested with sodium amalgam and water, and then acidified with HCl, is reconverted into phenol, which may be recognized by its odour.
2. Gently heated with *ammonia*, and a drop of *sodium hypochlorite* added, a deep blue colour (green in dilute solutions) is produced, changed to red by acids.
3. *Ferric chloride* gives a violet colouration; (not so sensitive as with salicylic acid —1 in 3000).
4. Phenol added to a mixture of *potassium nitrite* and *strong sulphuric acid*, gives a brown colour changing to green and finally to blue.

ANILINE.

Phenylamine. $C_6H_5NH_2$.

Oily liquid, colourless when pure. Boils at 183°·7 C. Slightly soluble in water. Easily soluble in alcohol, ether, and chloroform.

Solutions of aniline or its salts give with *bleaching powder* solution, not in excess, a violet or purple colouration, soon changing to brown.

Heated with *chloroform* and alcoholic solution of *caustic potash*, characteristic odour of phenyl carbamine C_6H_5NC is evolved. (See Chloroform.)

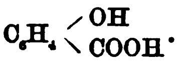
Treated with *sulphuric acid* and a fragment of *manganese dioxide* (or potassium bichromate) added, a blue colouration is obtained, changing to black after a time.

Heated with *potassium nitrite* and dilute H_2SO_4, phenol is formed, which may be recognized by its odour, &c.

$$C_6H_5NH_2 + HNO_2 = C_6H_5OH + H_2O + N_2.$$

SALICYLIC ACID.

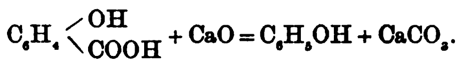
Orthoxybenzoic Acid.



$C_6H_4\begin{matrix}\diagup OH\\ \diagdown COOH\end{matrix}$.

White granular powder or prismatic crystals. Nearly insoluble in cold water. Soluble in hot water, alcohol, ether, or alkalies.

1. Heated with *lime*, it yields phenol, which may be recognized by its odour, &c.



$$C_6H_4\begin{matrix}\diagup OH\\ \diagdown COOH\end{matrix} + CaO = C_6H_5OH + CaCO_3.$$

2. *Ferric chloride* gives a fine violet colour even in *very* dilute solutions (1 in 100,000). The colour is destroyed by acids or alkalies.

3. *Bromine* water gives a white ppt. similar to that produced by phenol, and which behaves similarly with sodium amalgam; but the reaction is far less delicate.

BENZOIC ACID.

$$\begin{cases} C_6H_5 \\ COOH \end{cases}.$$

White feathery crystals. Characteristic odour especially on heating. Sparingly soluble in cold water. (The solubility is much increased by the presence of sodium phosphate.) Easily soluble in boiling water, alcohol, *ether*, and *chloroform*. Hydrochloric acid precipitates benzoic acid from strong solutions of its salts.

1. Heated with *lime*, benzene is evolved, which may be distilled off and identified as usual,

   $$C_6H_5 . COOH + CaO = C_6H_6 + CaCO_3.$$

2. *Heated alone* to about 120° C. it melts, and afterwards sublimes, giving an irritating vapour which readily condenses to white crystals.

3. Neutral *ferric chloride* gives, in neutral solution, a reddish-white ppt. of basic ferric benzoate $Fe_2(C_6H_5CO_2)_6 . Fe_2O_3$.

4. *Ammoniacal barium chloride*, in presence of alcohol, no precipitate.

HIPPURIC ACID.

Benzamidacetic Acid. $\begin{cases} CH_2NH(C_6H_5CO) \\ COOH \end{cases} = C_9H_9NO_3.$

Shining prismatic crystals. Nearly insoluble in cold water, *ether*, and *chloroform*. Readily soluble in boiling water, and in alcohol.

1. Boiled for some time with strong *hydrochloric acid*, it takes up the elements of water, forming amidacetic acid (glycocol) and benzoic acid—

   $$\begin{cases} CH_2NH(C_6H_5 . CO) \\ COOH \end{cases} + H_2O = \begin{cases} CH_2NH_2 \\ COOH \end{cases} + \begin{cases} C_6H_5 \\ COOH \end{cases}.$$

   The latter is mostly given off in vapour; and the former may be identified by adding to the solution an excess of *caustic potash* and a drop of *copper sulphate*, when a deep blue solution is obtained, which is unaltered by boiling.

2. *Heated alone*, it melts, and evolves aromatic odours, due to benzonitrile $C_6H_5 . CN$, benzoic acid, &c. Heated with lime it evolves benzene.

SUCCINIC ACID.

$$\left\{\begin{matrix}CH_2COOH\\CH_2COOH\end{matrix}\right.\cdot$$

Colourless crystals, easily soluble in water, alcohol, or ether; insoluble in chloroform. Is not precipitated from solutions of its salts by hydrochloric acid.

Heated alone, it gives off a suffocating vapour, easily condensing to crystals. Strongly heated in air it burns with a blue flame.

Ammoniacal barium chloride gives, in presence of alcohol, a white ppt. of barium succinate, $\left\{\begin{matrix}CH_2CO_2\\CH_2CO_2\end{matrix}\right\rangle Ba$.

Neutral ferric chloride gives, in neutral solutions, a brownish-red ppt. of basic ferric succinate, $Fe_2\left(\begin{matrix}CH_2CO_2\\CH_2CO_2\end{matrix}\right)_3 . Fe_2O_3$. (This ppt. when boiled with ammonia, gives ammonium succinate, which may be identified by the $BaCl_2$ test as above.)

PROTEIDS.

Approximate composition, $C_{72}H_{112}N_{18}O_{22}S$.

General characters:

Amorphous. Solutions lævorotatory.

Diffuse with very great difficulty (except peptones, which are highly diffusible).

General tests:

1. *Millon's reagent** gives a precipitate which turns red on heating. The liquid also turns red. With minute quantities a red solution only is produced.
2. *Strong nitric acid* produces a yellow colour, changed to orange by ammonia.
3. Heated with *caustic potash* and a few drops of *cupric sulphate* a violet solution is obtained. (Peptones give a red colour, if very little copper salt be added.)

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* Prepared by dissolving mercury in an equal weight of strong nitric acid at a gentle heat, diluting with twice its bulk of water, and allowing it to settle.

PROTEIDS (*continued*).

Egg-Albumin.

Soluble in water.

Silver nitrate, *mercuric chloride*, and *lead acetate*, precipitate it from its solution.

Strong alcohol first precipitates, afterwards coagulates it.

Strong acids, especially *nitric*, coagulate it.

Heated to about 70° C. it is coagulated. If however a dilute acid or an alkali be first added, no coagulation occurs on heating, and the solutions contain "acid-albumin" and "alkali-albumin" respectively, which are precipitated on neutralizing the liquid.

Egg-albumin differs from serum-albumin in some respects, *e.g.* in being coagulated by ether, and in its coagulated form being nearly insoluble in strong nitric acid.

Casein.

Insoluble in water, and in neutral saline solutions.

Soluble in dilute acids and in alkalies, reprecipitated on neutralization. If an *alkaline phosphate* be present, however, it is not reprecipitated on neutralizing its *alkaline* solution.

Casein very closely resembles alkali albumin in properties; according to some authors it is identical with potassium albuminate.

Fibrin

Has a filamentous structure, and possesses great elasticity.

Insoluble in water. Very sparingly soluble, and with change, in dilute alkalies, and in strong neutral solutions of sodium chloride, potassium nitrate, &c.

Fibrin has the property of readily decomposing hydrogen dioxide, oxygen being evolved. If fibrin be treated with *tincture of guaiacum*, and *hydrogen dioxide* added, a blue colour is obtained.

GELATIN or GLUTIN.

Amorphous. Percentage composition differs but slightly from that of the proteids. Easily soluble in water on heating, gelatinises on cooling. Aqueous solution is lævorotatory, and is not diffusible.

Tannic acid throws down a yellowish precipitate, even from very dilute solutions.

Mercuric chloride also precipitates it.

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